

**HERE'S HOW YOU  
CAN DO IT  
IN  
CHEMISTRY**

**Section 2.**

# **HERES HOW YOU CAN DO IT IN CHEMISTRY**

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**THE EXTRACTION OF USEFUL SUBSTANCES FROM THE EARTH (SEN 1979, Vol. 28 No. 1)**  
Dr G.W. Thickett, Belmore BHS

The following article is part of an address delivered at an inservice conference held at Belmore Boys' High School. This extract is a developmental sequence for a Year 7 chemistry unit which has been developed on the theme of extraction of useful substances from the earth.

**Extraction of Useful Substances from the Earth**

<b>Period</b>	<b>Major Idea</b>	<b>Developmental Sequence</b>
1,2	Extraction of metals from natural ores	<ol style="list-style-type: none"><li>1. Certain substances are found in their native state in the earth. Gold and silver.</li><li>2. Fascination of people with gold and silver from earliest times - used in ornaments.</li><li>3. Recognition by ancient tribes of copper and copper bearing ores. Extraction of these ores using heat and charcoal.</li><li>4. Lab experiment on the extraction of copper from copper carbonate on a carbon block.</li></ol>
3.	Physical properties of copper	Students discover the following properties of copper: <ol style="list-style-type: none"><li>1. colour, lustre</li><li>2. malleability, ductility, hardness</li><li>3. conductivity of heat and electricity.</li></ol>
4,5	Chemical properties of copper	Students examine some chemical properties of copper: <ol style="list-style-type: none"><li>1. not attacked or corroded by hot water or cold water</li><li>2. forms a black, scaly coating when heated in a bunsen flame</li></ol>

3. copper colours a bunsen flame green.

Properties (both physical and chemical) are summarised - students made aware of how these properties make Cu useful for water pipes and electrical wiring.

6,7 Preparation of copper oxide, carbon block decomposition of black oxide to Cu

Students heat strips of Cu sheet in a bunsen and scrape off and collect black scale. Process is repeated to obtain sufficient oxide for use on the carbon block. The black material is decomposed back to Cu on a carbon block. Students summarise these ideas:

copper gas ( $O_2$ ) combines

copper oxide forming black scale

8,9 Iron in the natural state

Meteoric iron. Ores containing iron. Colours of ores. Early people discover how to extract iron - charcoal and high temperatures. Iron ores in Australia

- mining sites
- processing sites.

10,11 Can iron ore be decomposed into iron in the lab?

Students attempt to reduce  $Fe_2O_3$  to Fe on a carbon block. By their failure, they see the requirement for higher temperature.

The blast furnace

Blast furnace operation examined simply along the following lines:

- temperature required
- need for coke
- limestone removes unwanted wastes from ore.

12 Properties of iron (1)

Physical properties examined as per copper:

- lustre, colour, malleability, ductility
- electrical/heat conductivity
- comparison of heat conductivity of iron and copper

13,14, 15	Properties of iron (2)	<p>Chemical tests on iron:</p> <ol style="list-style-type: none"> <li>1. effect of cold/hot water over <ol style="list-style-type: none"> <li>a) the short term</li> <li>b) the long term</li> </ol> </li> <li>2. experiment set up to show the requirement for air and water in rusting</li> <li>3. burning of iron wool to produce a grey-black substance - students reminded of a similar effect with copper. New substance is an iron oxide.</li> </ol>
16	Removal of oxygen from the air during rusting	<p>Moist steel wool removes 20% of the air as it rusts (as revealed by a rise in water level when the experiment is conducted in a closed tube over water. As the iron rusts, a type of iron oxide forms:  <math>\text{iron} + \text{gas in the air} \rightarrow \text{iron oxide}</math>  Students encouraged to see that air contains 20% by volume of a reactive gas called oxygen.</p>
17	Combustion - the experiments of Priestley and Lavoisier	<p>Rusting experiment compared with Priestley's and Lavoisier's experiments with mercury and mercury calx. Burning involves combination of the metal with 20% of the air (oxygen). Ores of mercury (cinnabar) mentioned.</p>
18	Summary of ideas	<p>Students revise the following ideas</p> <ol style="list-style-type: none"> <li>a) metals can be extracted from their ores by heating; heating can cause the ore to break down to release the metal, under certain conditions</li> <li>b) mercury and copper are released more easily from their ores than iron</li> </ol>
19	Summary of ideas (continued)	<ol style="list-style-type: none"> <li>c) metals can be made to react with the oxygen in the air to produce new substances called oxides; some metal oxides are</li> </ol>

		found naturally as ores (eg. iron oxide in iron ore)
		d) metal oxides can be decomposed back to the metal and oxygen under certain conditions; some oxides (such as mercury oxide) are more easily decomposed than others (eg. iron oxide).
20,21	Methods of extraction of naturally occurring substances without changing them	Salt is reviewed as being of major importance to all - now and in the past; salt valued and used as money, salt required for our diet; mining of salt; salt extraction from the sea; large scale methods.
22,23	Laboratory evaporation of sea water	Students study the techniques involved in evaporation of salt water; names of equipment; reasons behind evaporation technique involving separation on the basis of differing BPs; crystals form; fast and slow evaporation.
24,25	Distillation to obtain fresh water	Concept of distillation, involving evaporation and condensation; apparatus required; students distil salt water, collect distillate and evaporate a little distillate on glass slide to check that no salt is present ((do a control with a drop of salt water).
26,27	Distillation of water containing mud and salt	Students note that both the mud and salt remain behind in the distillation flask, only the low BP water is distilled; large scale distillations in industry, eg. petrol and wine; ideas of fractional distillation based on differing BPs
28,29	Filtration	Ideas of solubility as a criterion for filtration; industrial examples of uses of filtration; students filter a suspension of iron oxide

in water; equipment and techniques examined.

30,31 Ideas of insolubility

1. Students test the solubility of each of the following in water:
  - copper oxide, iron oxide, sugar, bicarbonate of soda.
2. On the basis of solubility results students design an experiment to separate a solid mixture of sugar and copper oxide.
3. Experiment is performed using the techniques of filtration and evaporation; methods of growing large crystals examined.

32 Review

Students review the main ideas of the unit:

1. We obtain useful substances from the earth via -
  - a) chemical decomposition methods, eg. heating, heating with carbon; some ores are more easily extracted than others, eg. cinnabar and malachite are more easily decomposed than iron ore
  - b) physical separation methods (which do not alter the material), eg. evaporation, distillation, filtration.
2. Metals when heated with oxygen of the air combine with it to form new substances called metal oxides. Metal oxides can be decomposed under certain conditions to form the metal once more. The oxygen is then driven off.



# **PRACTICAL EXAMINATION: IDENTIFICATION OF AN UNKNOWN COMPOUND** (SEN 1979, Vol. 28 No. 4)

Geoffrey Thickett, Belmore BHS

## **INTRODUCTION**

The following experiment is useful as both a practical examination and an exercise in developing manipulative proficiency. CYCLOHEXANE is the best unknown to use with its melting and boiling points being sharp and readily measurable in a school laboratory. Benzene is NOT recommended owing to its toxicity. Other alternatives include 2-methyl 2-propanol and cyclohexanol.

## **Aim**

To identify an unknown organic compound by determining its density, melting point and boiling point.

## **Background**

In this exercise it is vital that the determination be performed accurately. Once the data has been collected, reference is made to the accompanying table of chemical data in order to identify the unknown.

## **Procedure**

### **A. Density Determination**

1. Determine the mass of a clean, dry 10mL measuring cylinder on a sliding poise balance. Record this mass.
2. Remove the measuring cylinder from the balance pan and add (with the aid of a dropper) the unknown liquid to the cylinder until the volume is exactly 10.0mL (bottom of meniscus). (V)
3. Determine the mass of the cylinder plus the unknown liquid. Thus calculate the mass of the 10.0mL of the unknown. (m)
4. Calculate the density of the unknown liquid using the formula  
$$D = m/V \text{ (g.mL}^{-1}\text{)}$$

### **B. Boiling Point Determination**

1. Set up the quickfit apparatus as directed and ensure that there is no strain placed on the joints. The distillation flask is heated by a surrounding water bath. The unknown is highly flammable and bunsen flames **must not** be brought near it or its vapours. Ensure that water is flowing through the condenser jacket throughout the distillation.
2. Pour the 10mL of unknown from Part A into the distillation flask and use your hot water bath as a source of heat. The thermometer bulb should be about 1cm above the surface of the boiling liquid. Distil at a constant rate and record the thermometer reading every minute from the beginning of the experiment until distillation is

complete. Remember that a constant temperature reading is an indication of the boiling point.

3. Plot a graph of temperature versus time and determine the boiling point from the graph.

### C. Melting Point Determination

1. Insert a thermometer in the test tube of distillate collected in Part B. Place the tube in an ice water bath or refrigerator until the unknown has completely solidified and the thermometer reads 2°C or less.
2. At time zero, remove the tube from the ice bath and place it in a test tube rack to warm up. Measure and record the temperature every minute until the temperature rises about 5°C above the melting point. Return the unknown liquid to the front bench. Do not discard.
3. Plot a graph of temperature versus time and indicate on the graph the melting point of the unknown.

### Treatment of Data

Use the three pieces of experimental data to identify the unknown from the list of possibilities supplied. In your report you should list all the possibilities and systematically eliminate them, giving your reasons. Remember that all your data may not be as accurate as you think. Name the unknown, give its molecular formula and draw its graphic formula.

### DATA SHEET

Name	Density (g/mL)	MP (°C)	BP (°C)
Hexadecane $C_{14}H_{34}$	0.774	18.52	87.5
Tribromomethane $CHBr_3$	2.890	8.3	149.6
Dibromomethane $C_2H_4Br_2$	2.180	10.0	131.7
2-methyl-2-propanol $C_4H_{10}O$	0.786	25	82.5
3-methyl-2-butanol $C_5H_{10}O$	0.818	171	12.9
Cyclohexanol $C_6H_{12}O$	0.962	25.5	161.1
Cyclohexane $C_6H_{12}$	0.779	6.5	80.7
Methanoic acid $CH_2O_2$	1.2203	8.4	100.9
Benzene $C_6H_6$	0.879	5.5	80.1
Ethyl acetate $C_4H_8O_2$	0.901	-83.6	77.2
Ethanol $C_2H_6O$	0.789	-114.5	78.5
Cyclohexene $C_6H_{10}$	0.810	-103.7	83.3
2-propanol $C_3H_8O$	0.785	-89.5	82.4
Butanone $C_4H_8O$	0.815	-86.9	79.5
Nitrobenzene $C_6H_5NO_2$	1.203	5.7	210.9

# PREPARATION OF ETHANOL BY FERMENTATION (SEN 1980, Vol. 29 No. 2)

Rees Bunker, Heathcote HS

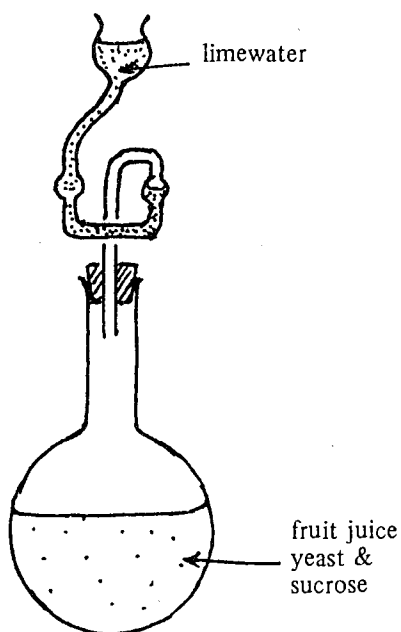
## Introduction

When grain, tubers and fruit ferment, the carbohydrates in them are converted by a series of reactions into ethanol,  $\text{CH}_3\text{CH}_2\text{OH}$ . Various enzymes contained in yeast speed up the reactions involved.

## Method

To a 370mL can of pure fruit juice, add 20g of sucrose and one packet of dried yeast or 2g of compressed yeast. Put this mixture into a 500mL flask sealed with a modified thistle funnel as shown in Figure 1, or with a piece of rubber tubing submerged in a beaker of water. Allow to stand for 2-5 days in a warm atmosphere. the temperature should be 25-35°C.

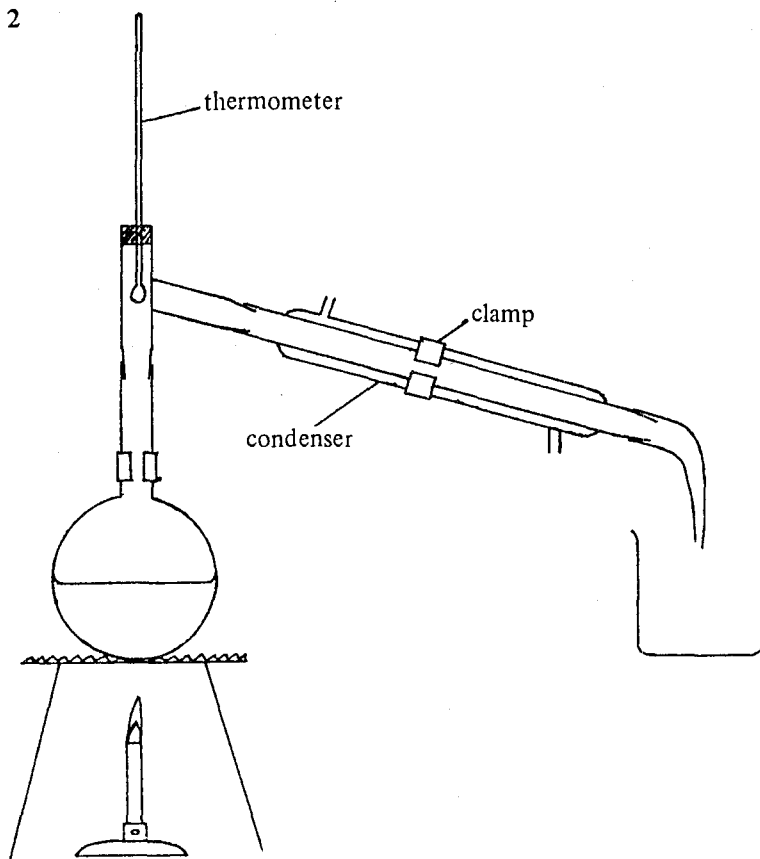
FIG. 1



Decant the liquid without disturbing the yeast and filter through cotton wool if required. Carefully distil this aqueous solution as shown in Figure 2. **Caution:** much frothing may occur at first, so heat cautiously. Note the temperature at which the distillate passes over.

Collect the distillate until the temperature rises above 97°C. Place the distillate in an evaporating basin and ignite with a burning splint. What is the colour of the flame? If the distillate does not ignite then redistill it with the temperature reaching no more than 85°C. Remove any remaining water by allowing to stand overnight in a stoppered flask containing some calcium oxide.

FIG. 2



### Questions

1. Why is it important to control the temperature?
2. What type of gas is evolved by the fermentation process?
3. What properties of ethanol were you able to observe?
4. Comment on any differences between the odours of the first and second distillates (if prepared).
5. Write an overall equation for the reaction of the sucrose  $C_{12}H_{22}O_{11}$  in the formation of ethanol.
6. What is the equation for the burning of ethanol?
7. Describe some important industrial uses of ethanol.
8. Alcohol is a prime factor in a major portion of cases of human death and misfortune, for example road accidents, sclerosis of the liver, crime and violence, marital disharmony and family breakdown. In the 1920s the United States government introduced prohibition of alcohol for a period in an attempt to reduce the harmful effects of alcohol on the community. Consider the possible effects of a similar prohibition in Australia. Summarise the arguments for and against such a step.

While performing the well known demonstration of the differing expansion coefficients of water and aniline recently, I thought of a new approach.

Instead of adding the cold aniline from the burette to cold water in a 500mL beaker, watching it sink and then heating with a bunsen burner to observe the magic temperature when the densities of the two liquids becomes the same and the spherules hang weightless, I thought out the following variations which do not require the heating of aniline.

Add a drop or two of aniline to a 500mL beaker, half filled with water and then dissolve sufficient sodium chloride to make the aniline float. Now carefully pour the cold water onto a floating cork in the beaker, thus establishing a density gradient up the beaker, the aniline spherules, of course, now hang weightless at the level where their densities are the same as salt water.

Now adjust the burette orifice to the level of the suspended spherules and carefully open the tap. A weightless aniline sphere now grows and more aniline can be added to the burette if required.

Using this method I have grown spherules up to 100mm in diameter before running out of aniline, which is removed afterwards using a separating funnel.

On completion of the above, which takes about five minutes, class discussion is almost limitless, covering density, Archimedes' Principle, weightlessness in space, surface tension, the shape of the earth (the 'sphere' is not in fact spherical), the solubility of so called immiscible liquids, expansion coefficients and Stokes' Law (viscosity).

All the students to whom I have shown this demonstration have been fascinated and I have even been offered money to buy more aniline and hence produce even larger spheres!

(Reprinted from SEN 1970, Vol. 19 No. 3.)

## **A COLOURED CLOCK REACTION (SEN 1980, Vol. 29 No. 3)**

A.J. Sperring and R.C. Warren, Sydney Teachers' College

### **Concepts: Uses**

Rates of reaction; stepwise reactions; change of pH during a reaction; as a colourful demonstration during science fairs, open days, etc.

### **Materials**

Sodium metabisulfite - 20g  
sodium sulfite (anhydrous) - 3g  
formalin - 90mL  
indicators e.g. phenolphthalein  
universal indicator  
250mL beakers - two or more  
white background.

### **Preparation**

- \* Solution A: 20g  $\text{Na}_2\text{S}_2\text{O}_5$   
3g  $\text{Na}_2\text{SO}_3$   
1000mL water.  
Dilute this solution by four to give Solution A.
- \* Solution B: 90mL formalin  
910mL water.

### **Procedure**

1. Mix equal volumes of Solutions A and B thoroughly, after adding about 2mL of indicator solution to one of them.
2. Stand the mixture against a white background.  
As a variation, or in studies of the effect of temperature on reaction rate, the above procedure can be repeated after heating one or both of the solutions in a water bath. Heating one solution to about 20°C above room temperature approximately doubles the rate of reaction.

### **Observations**

If the two solutions are mixed thoroughly, a homogeneous colour change should occur after an initial induction period. With phenolphthalein the colour change is from colourless to red; with universal indicator a sequence of colour changes occurs, at a varying rate.

With insufficient mixing the colour change occurs in splotches (itself an arresting phenomenon).

The colour change is not as sharp as in the familiar iodate-sulfite clock reaction, though it is still reasonably rapid.

## Comments

The reaction mechanism is:

1.  $\text{HCOH} + \text{HSO}_3^- \text{ --- slow } \longrightarrow \text{CH}_2\text{OSO}_3^-$
2.  $\text{HCHO} + \text{H}_2\text{O} + \text{SO}_3^{2-} \text{ --- fast } \longrightarrow \text{CH}_2\text{OSO}_3^- + \text{OH}^-$
3.  $\text{OH}^- + \text{HSO}_3^- \text{ --- slow } \longrightarrow \text{SO}_3^{2-} + \text{H}_2\text{O}$
4.  $\text{OH}^- + \text{indicator} \text{ --- fast } \longrightarrow \text{colour change}$

Colour change is due to the production of  $\text{OH}^-$  and its reaction with the indicator. However,  $\text{OH}^-$  is not 'available' for reaction with the indicator until the  $\text{HSO}_3^-$  has been used up.

## THE SIZE OF A MOLECULE (SEN 1980, Vol. 29 No. 4)

R.P. Jones, Macintyre HS

### Aim

To determine the order of magnitude of the size of a molecule.

### Method

Take a **very** large tray. the wooden tray from a laboratory trolley is only just big enough. Much better, but requiring a little more sense of adventure, is to use the vinyl-tiled floor of the laboratory and build a circular plasticine dam wall about one metre in diameter. Now pour sufficient water in the tray or within your plasticine dam to fill it to about 1cm depth. Now wait awhile. Before the next step the water must be perfectly still. (If you think this is crazy so far, wait till I tell you the next step!)

From a great height, very lightly dust the surface of the water with talcum powder - no, don't overdo it - you only need sufficient talcum dust to make the surface of the water visible.

Now take the thinnest whisker from the laboratory cleaner's straw broom! (Yeah, you're convinced I'm mad - but hang in there - this really works!) This is the tricky part. Dip the fine straw into a bottle of oleic acid and withdraw it at just the right speed so that, when clear of the bottle, just **one** small drop of oleic acid falls off the end of the straw. (This takes some skill, we don't want a stream of drops, just **one**) When you have mastered this technique (in front of the class), repeat it over the centre of the dusty water so that just one tiny drop of oleic acid falls onto the surface.

ZAROOM!!! the effect is mind boggling!

### Calculations

All I am going to tell you is that the volume of a drop 1mm in diameter equals the volume of a circular monolayer. You take it from there.

### Comments

This is a spectacular experiment and the calculations yield about  $10^{-9}\text{m}$  for the size of a molecule, which is right on target. If you have never seen it done before, then go for a dummy run before you spring it on the class. Oh, and by the way, if you want to repeat it you must start with fresh water and talcum all over again. You can't use the same trayful.



# AVOGADRO'S NUMBER: DETERMINATION USING A MONOLAYER

(SEN 1982, Vol. 31 No. 2)

Di Herford, St. Claires College, Waverley

Experimental details are outlined in *Chemical Science Teachers Manual* p.14 (and above). Instead of using ethanol as the solvent, ether may be used as the solvent for oleic acid. Here is an outline for the calculations, using the results of some students.

Molar mass of oleic acid	= <u>282</u>
Mass of 1mL of oleic acid	=0.82g
∴ number of moles/mL of <b>pure</b> oleic acid	= <u><math>2.91 \times 10^{-3}</math> moles/mL</u>
<b>Diluted oleic acid solution</b> 54 drops	= <u>1.2mL</u>
∴ volume of one drop	= <u><math>2.22 \times 10^{-2}</math> mL</u>
Dilution of oleic acid	= <u>1 in 20</u>
∴ volume of <b>pure</b> oleic acid used	= <u><math>1.11 \times 10^{-3}</math> mL in 1 drop</u>
Number of moles of oleic acid used	= <u><math>3.23 \times 10^{-3}</math> moles</u>
Volume of oleic acid used will be the same as the volume of the monolayer.	

## Volume of the monolayer:

Diameter of the monolayer	= <u>43cm</u>
Radius of the monolayer	= <u>21.5cm</u>
Assume the monolayer has the shape of a cylinder, $V = \pi r^2 h$	
Diameter of one molecule	=height of cylinder
	$h = V / \pi r^2$
	= <u><math>7.46 \times 10^{-7}</math> cm</u>
Radius of one molecule	= <u><math>3.82 \times 10^{-7}</math> cm</u>
Volume of one molecule	= $4/3 \pi r^3$
	= <u><math>2.335 \times 10^{-19}</math> cm<sup>3</sup></u>
∴ number of molecules in the monolayer	= <u>volume of monolayer</u>
	volume of one molecule
	= <u><math>1.11 \times 10^{-3}</math></u>
	$2.33 \times 10^{-19}$
	= <u><math>4.75 \times 10^{15}</math></u>
Number of molecules/mole	= <u><math>4.75 \times 10^{15}</math></u> = <u><math>1.47 \times 10^{21}</math></u>
	$3.23 \times 10^{-6}$

## **A CHEAP SELECTIVE-ION ELECTRODE (SEN 1981, Vol. 30 No. 1)**

Iain Townsend, Mitchell CAE

### **Summary**

Selective-ion electrodes are widely used in industry and in research. However, they are unlikely to find their way into the high schools until some way can be found to decrease their price. This article describes a way to use a broken pH electrode as a selective-ion electrode, thus killing two birds with the one stone. A glass membrane electrode is such a fragile thing that most of us have had one on our hands at one time or another! However, the broken electrode and its attendant pH meter can now be used in a variety of analyses in the environment.

### **Introductory Theory**

The selective-ion electrode which will be most familiar to the majority of readers will be the glass electrode used in pH measurements. The glass electrode is sensitive to the activity of  $H^+$  in solutions and, in conjunction with the saturated calomel electrode, forms an electrochemical cell the output of which can be readily measured. The sensitivity of the glass electrode is due to the ability of the glass membrane to exchange  $H^+$  ions for alkali metal ions in the glass. If this membrane is subsequently broken and removed the silver electrode is exposed directly to the solution. The electrode is coated with an adherent coat of silver chloride. Fricke and Kuntz (1977) state that such an electrode will be in solubility equilibrium with the chloride ion activity present in the solution. The voltage output of such a cell, measured using the mV function of the pH meter, will obey the equation:  $E = 2.3 RT/nf \log A$

Where  $E$  is the potential,  $A$  is the ionic activity,  $n$  is the valency and  $R$  and  $F$  have their usual meanings. The electrical potential of the cell is the reduction potential of that activity of  $Ag^+$  ion which is present in solution in equilibrium with the chloride ion and in the presence of  $AgCl$  precipitate.

Figure 1. shows a plot of mV vs  $-\log Cl^-$ ; Fricke and Kuntz claim that a double junction reference electrode is necessary for most ions analysed by this technique; however this was not done since the broken glass electrode used had its saturated calomel electrode as an integral part. Stopping the leakage of chloride ions into the solution being tested would have been impossible. In spite of this good results were obtained. A linear regression was carried out on the data, using a Hewlett-Packard HP97 calculator. Results showed that the data were highly linear ( $r^2 = 1.00$ ). It is likely that for the concentrations employed and the level of accuracy, this refinement can be omitted. Furthermore, most readers would only be interested in selective ion

electrodes as teaching experiments where absolute accuracy may be desirable but not strictly necessary.

## EXPERIMENTAL

The pH meter employed was a Pye Unicam Model 292 and the electrode was a Pye-Ingold Eo7 combined glass and reference electrode. The electrode had previously been dropped and so it was necessary to trim the jagged glass off, taking care not to disturb the Ag/AgCl electrode or the reference electrode. The electrode was stored in distilled water when not in use. It is probable that any meter/electrode combination could be used, provided the meter measures  $\pm 700$  mV, estimating the last place visually.

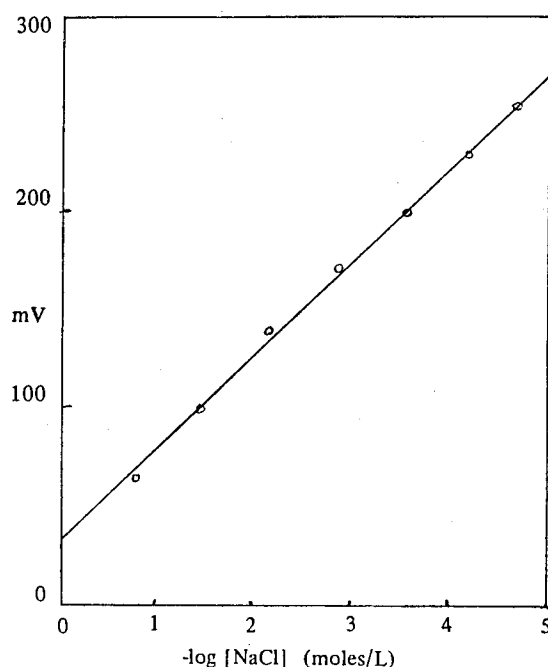


Figure 1: Plot of voltage output for different concentrations of sodium chloride in distilled water. The line of best fit was obtained from a linear regression as described in the text.

## Calibration

The calibration curve shown in Figure 1 was obtained by preparing a standard NaCl solution (0.1737M) and then 1:5 serial dilutions. Set the meter to measure mV and follow the manufacturers' instructions as to calibration, etc. In my case, the meter was set at zero for distilled water. Solutions of NaCl prepared as above were then used, starting at the lowest concentration. A magnetic stirrer can be used, but is not necessary. Allow at least one minute for the reading to become steady.

It is necessary to recalibrate before each use. Discard the standards whenever the calibration is no longer a straight line. The lowest concentrations appear to be most affected either due to the effect already described by Fricke and Kuntz or else by contamination.

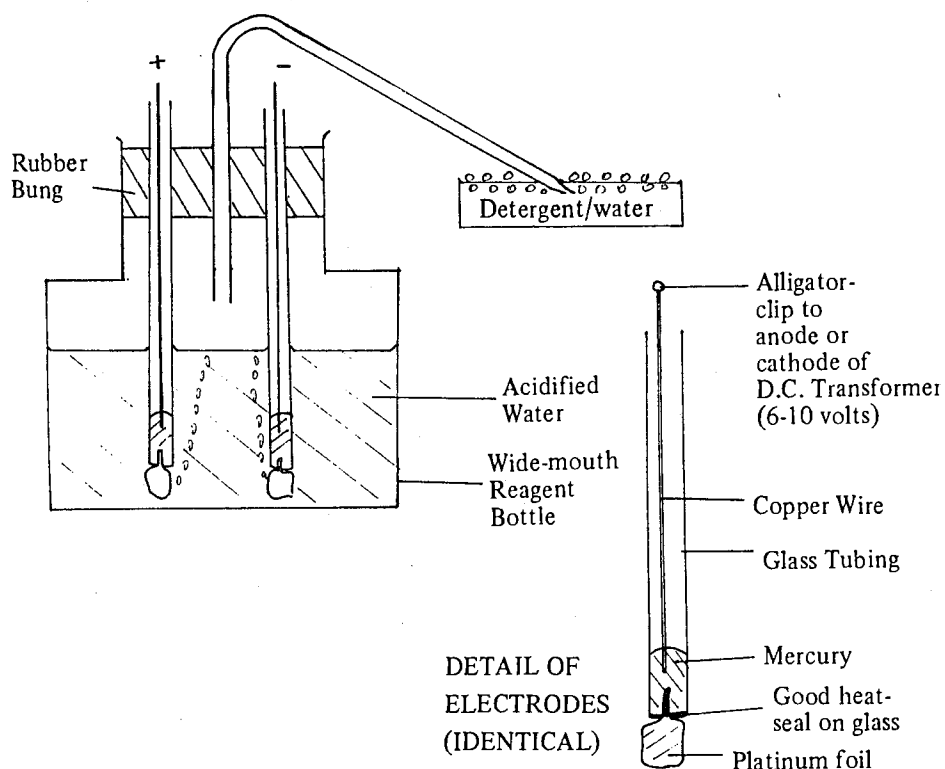
The electrode is now ready for use in analysing water samples for their chloride content. For chlorides, interferences from the other halides are the most troublesome, but in many natural waters the heavier halides at least should be present in insignificant concentrations. Solutions should not be strongly acidic or basic.

#### REFERENCE

1. Fricke, G.H. and Kuntz, M.J., *Journal of Chemical Education*, 54, 1977 517-520.

For many years now I have used the simple apparatus described below to demonstrate the energy potential of hydrogen gas.

The apparatus is basically similar to any 'electrolysis of water' apparatus. However, in this case, both gases produced (2 volumes hydrogen, 1 volume oxygen) are collected **together**, thus they are in the correct molar proportions for recombination to form water. Unlike the normal combustion of hydrogen in air (21% oxygen), this reaction results in the immediate release of **all** the energy available in hydrogen gas.



The hydrogen/oxygen gas mixture is bubbled into a petri dish of detergent and water. When a good collection of bubbles has been produced, **turn off the current and remove the apparatus to a safe distance**. A match is then applied to the bubbles and a safe, open explosion like a rifle shot is produced.

Students may be asked to trace the energy pathway (electrical energy - chemical potential energy - kinetic energy). They may discuss the

efficiency of the process (some energy lost as sound; also the apparatus becomes warm after a period).

The reaction can be related to the current discussion concerning hydrogen powered cars. Students can be asked the origin of the energy needed to produce the hydrogen and oxygen gases (the sun).

A recent television program, 'Hydrogen - the Forever Fuel', may be available on video as a follow-up teaching aid.

**Note on Construction**

The heat seal between the glass tubing and the platinum foil must be perfect. If it is not the mercury conductor will be rapidly lost.

**Concepts: Uses**

Motivation; formation of a gel; solubility of salts.

**Materials**

Calcium acetate - 34g/100mL; ethanol (or methylated spirits) - about 250 mL; 3 beakers.

**Safety Notes**

Care with ethanol

**Preparation**

- \* Prepare a saturated solution of calcium acetate (34g/100mL). An appropriate volume would be 100mL. Place this in a 500mL beaker (or larger).
- \* Place about 250mL of methylated spirits (or ethanol) in a beaker.

**Procedure**

- \* Quickly pour the methylated spirits into the solution of calcium acetate.
- \* Then attempt to pour the contents of this beaker into another beaker.

**Observation**

- \* The calcium acetate should form a gel that will solidify.

**Comments**

- \* The calcium acetate and ethanol form a gel (a liquid dispersed in a solid), demonstrating that the calcium acetate is not soluble in ethanol (though soluble in water).
- \* The gel can be squeezed to remove most of the alcohol, placed on a heat resistant mat and lit (the alcohol will burn). This is sometimes called 'burning snow'.

## ENERGY FROM CHEMICALS (SEN 1981, Vol. 30 No. 2)

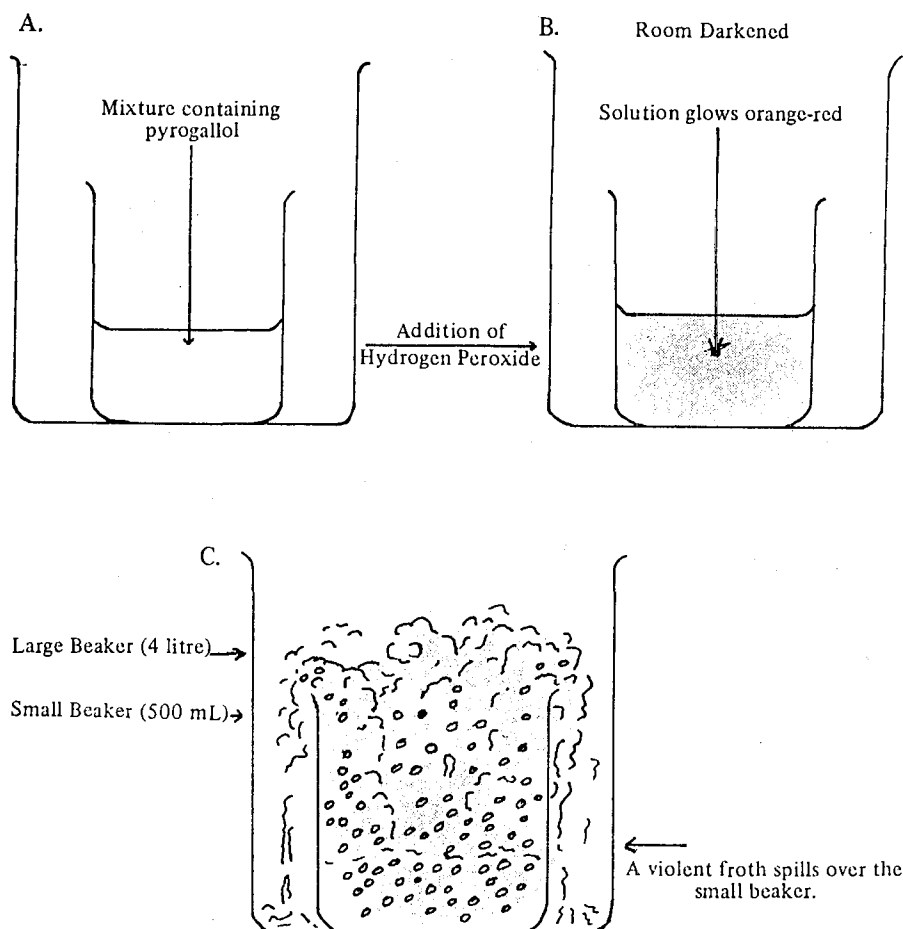
Tom Rozga, Sydney Technical HS

### Introduction

A flame is a zone in which chemical reactions between gases is accompanied by the evolution of heat and light. A flame could be simply described as glowing gas. Solids emit flames if gases or vapours form on heating. Transparent gases such as nitrogen and oxygen do not glow when heated to a high temperature within tubes.

Flame is produced in chemical reactions liberating much energy and the emission of light is due to chemical reactions. **Chemiluminescence** may be regarded as a cold flame. The glow of phosphorus is a familiar example and if ether is dropped on a hot iron plate a greenish phosphorescent flame is seen.

The following reaction demonstrates chemiluminescence in solution. Light of the wavelength emitted accelerates the reaction which is thought to involve the oxidation of pyrogallol and the evolution of much heat.





### **Preparation of Solutions and Procedure**

1. Add, in order, into a 500mL beaker: 10mL of 10% aqueous pyrogallol, 10mL of formaldehyde and 20mL of a saturated solution of potassium carbonate (21g in 20mL of water).
2. Place the beaker into a 4L beaker, darken the room and then add 30mL of 100 volume hydrogen peroxide. Stand well clear and observe the orange-red glow followed by a vigorous reaction which froths violently over the small beaker.

### **NOTE**

Make sure you do not spill any of the final product on yourself or the furniture. It is corrosive. Have pupils stand well clear from the demonstration.

100V hydrogen peroxide is not on the Requisition. You will have to purchase it from a chemical supplier and preferably obtain it fresh as it decomposes with time and the effectiveness of the demonstration will dwindle.

I have performed this demonstration for junior and senior chemistry classes. It provides an exercise for accurate observation as well as to demonstrate energy conversions in a spectacular manner.

An addition to the demonstration is to add 100mL of 0.1% luminol in 2M sodium hydroxide after the addition of 100mL of formaldehyde in Step 1. This causes the bubbling froth produced towards the end of the demonstration to glow a lilac colour and lighten a dark room. Luminol is expensive and may be obtained from Scientific and Research Equipment, Thornleigh. Approximate price \$25 for 5g, but this amount will last out many demonstrations.

### **Reference**

*General and Inorganic Chemistry*, Partington J.R., Macmillan, London, Fourth Edition, 1966.

**THE FOUNTAIN EXPERIMENT** (SEN 1981, Vol. 30 No. 2)  
R.G.Paton, Diploma in Education student, Sydney Teachers' College

**Concepts**

Air pressure, solubility of gases, ammonia gas as a base.

**Apparatus**

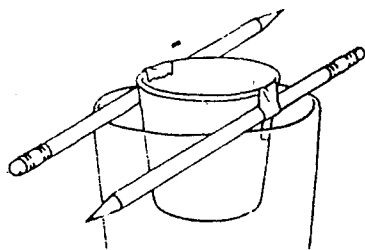


Figure 1a.

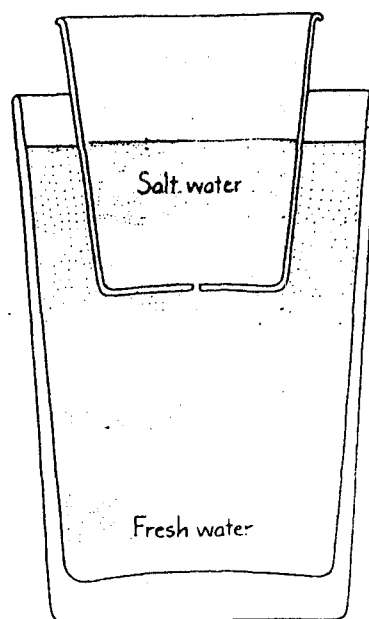


Figure 1b. *The basic salt oscillator*

**Generation of Dry Ammonia Gas**

Drop concentrated ammonia solution onto about 20g NaOH pellets in a generator as shown and collect by downward displacement of air.

**Generation of HCl Gas**

Add 10mL concentrated  $\text{H}_2\text{SO}_4$  to 20g NaCl solid in a generator as shown and collect by upward displacement of air.

**Safety Precautions**

Generation of gases should be carried out in a fume cupboard.

**Procedure**

Start the fountain by squirting water from the eye dropper into the florence flask to dissolve some of the gas and so lower the pressure inside the flask. Solution is forced into the flask from the beaker.

**Comments**

Both ammonia and HCl gases are extremely water soluble, hence the rapid drop in pressure inside the flask.

## **Alternatives and Additions**

1. A simple demonstration of air pressure/contraction of gases on cooling can be obtained by heating a small amount of water in a florence flask and while still hot, placing a rubber bung containing a long glass tube in the neck of the flask. The flask is inverted over a beaker of water as in the diagram above. Cooling of the gas inside the flask causes a 'fountain' similar to that above.
2. An inverted florence flask containing dry ammonia gas over a beaker of strong hydrochloric acid solution instead of water can be used to demonstrate the reaction of  $\text{NH}_3$  as a base and so initiate a discussion of 'What is a base?'

## **Reference**

Joseph et al., *A Sourcebook For the Physical Sciences*, Harcourt, N.Y., (1961), pp160-161.

## CATALYSIS, A TRANSITION METAL AND OXIDATION-REDUCTION

(SEN 1982, Vol. 31 No. 1)

Dennis Mather, Leichhardt HS

The following reaction is useful because it is directly relevant to three sections of the chemistry syllabus, but it could also be used for a junior chemistry demonstration as well.

Some catalysts provide a surface upon which a reaction takes place, others take part in the reaction forming an intermediate which subsequently decomposes to yield the original material. The evidence for such a process is normally difficult to provide without sophisticated equipment. An exception is the oxidation of potassium sodium tartrate (Rochelle salt) by hydrogen peroxide using cobalt (II) chloride.

### Procedure

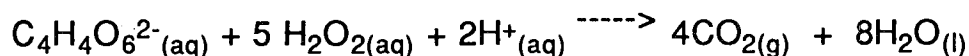
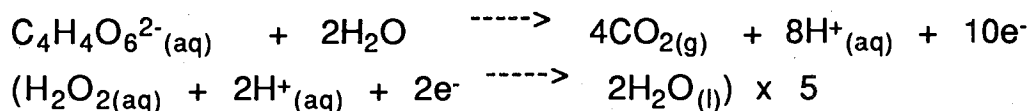
Weigh out 1g of potassium sodium tartrate ( $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ ) and place it in a beaker. Dissolve it in 25mL of water with gentle heating. When the solution reaches 60-70°C remove the bunsen, place the beaker on a piece of white paper on the bench and add 2mL (approximately) of 27% (100 volume) hydrogen peroxide (handle carefully, hydrogen peroxide will produce mild burns on the skin). Where more dilute hydrogen peroxide solutions are available the volume will need to be increased. At this stage little reaction will be observed.

Add 1mL (approximately) of cobalt chloride catalyst solution (10g per 100mL), stir and carefully observe:

1. the onset of the reaction
2. a change in colour as the catalyst forms and intermediate compound
3. a return to the original colour of the catalyst when the reaction is complete.

Tartrate is the conjugate base of a dicarboxylic acid - see Figure 1.

It is oxidised in the same way as oxalic acid:



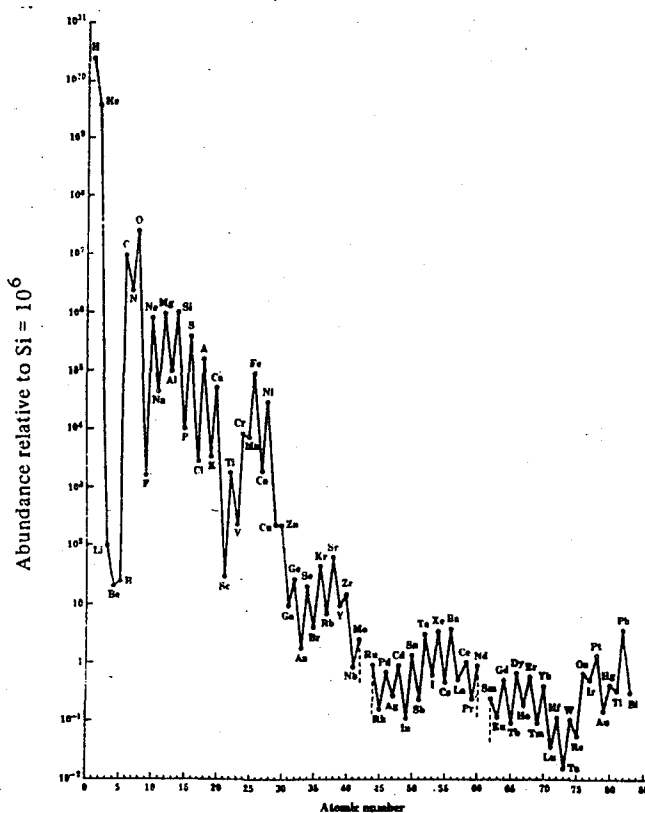


Figure 1. Comparative abundance of the elements in the universe relative to silicon =  $10^6$

From Distribution of Elements in Our Planet, Ahrens, McGraw-Hill, 1965

There is an initiation time which will depend on the concentration of cobalt (II) in the reaction mixture (see below), as well as ambient temperature, after which carbon dioxide is evolved as a gas. Simultaneously the solution changes colour from the pale pink of the aqueous cobalt (II) ion to a bright green. This colour probably indicates the presence of cobalt (III). When the effervescence ceases the green colour simultaneously reverts to the previous pink.

The reaction is quite sensitive to the concentration of cobalt (II) ions in a most unexpected way. If too much is added the reaction is not catalysed as well as it is with smaller concentrations. In addition the green colour of the reaction intermediate is not as apparent because it is mixed with the pink of the unchanged aqueous cobalt (II) ions.

The carbon dioxide evolved can be collected in a gas jar over lime water. The changes in the colour of the solution (pink to green to pink) demonstrate in a most explicit way the use of a catalyst in a reaction followed by its return at the end of the reaction.

# COMPARATIVE ABUNDANCES OF THE ELEMENTS (SEN 1982, Vol. 31 No. 1)

Dennis Mather, Leichhardt HS

It has been my experience that the new 2 Unit Chemistry syllabus has asked for information which was not required by the previous syllabus. Some of this information has been difficult to obtain while other data were found in references not usually used by chemistry teachers. For instance, in core unit (a) the last suggested experience states:

*Preparation of a chart showing the comparative abundances of the elements in the universe, on earth, in animal and plant bodies, and as used in industry*

I am still searching for the comparative abundances as used in industry!

The following table represents the information relevant to this suggested experience that I have gathered myself, from other teachers as well as from references.

Element	Crust	Element	Crust
Li	20	Ag	0.07
Be	2.8	Cd	0.2
B	10	In	0.1
F	625	Sn	2
Na	2.4%	Sb	0.2
Mg	1.95%	Te	
Al	8.2%	I	0.5
Si	28.2%	Cs	3
P	1,050	Ba	425
S	260	La	30
Cl	130	Ce	60
K	2.1%	Pr	8.2
Ca	4.2%	Nd	28
Sc	22	Sm	6
Ti	0.57%	Eu	1.2
V	135	Gd	5.4
Cr	100	Tb	0.9
Mn	950	Dy	3
Fe	5.6%	Ho	1.2
Co	25	Er	2.8
Ni	75	Tm	0.5
Cu	55	Yb	3
Zn	70	Lu	0.5
Ga	15	Hf	3
Ge	1.5	Ta	2
As	1.8	W	1.5
Se	0.05	Re	
Br	2.5	Os	
Rb	90	Ir	
Sr	375	Pt	
Y	33	Au	0.004
Zr	165	Hg	0.08
Nb	20	Tl	0.45
Mo	1.5	Pb	12.5
Ru		Bi	0.17
Rh		Th	9.6
Pd		U	2.7

Table 1. Estimated abundances of elements in the Earth's crust. Unless otherwise stated the values are in parts per million (1 ppm = 0.0001%)

From Distribution of the Elements in Our Planet, Ahrens, McGraw-Hill, 1965

Mineral		Annual output (million tonnes)
iron ore	Fe	675
manganese	Mn	18.36
aluminium	Al	7.8
copper	Cu	8.4
chromate	Cr	4.8
asbestos	—	3.5
lead	Pb	2.9
nickel	Ni	.5
tin	Sn	.2
molybdenum	Mo	.1

Table 2. The ten most commercially important minerals by value of annual output (source not known)

Element	Symbol	Approximate % (by weight) of man	Approximate % (by weight) of maize	Approximate % (by weight) of yeast
oxygen	O	65	75	72
carbon	C	18	13	14
hydrogen	H	10	10	10
nitrogen	N	3.3	0.45	2.4
calcium	Ca	1.5	0.07	0.01
phosphorous	P	1.0	0.06	0.5
potassium	K	0.35	0.28	0.6
sulfur	S	0.25	0.05	0.005
sodium	Na	0.24	trace	0.05
chlorine	Cl	0.19	0.04	—
magnesium	Mg	0.05	0.06	0.05
iron	Fe	0.005	0.03	0.005
manganese	Mn	0.003	0.01	—
silicon	Si	trace	0.46	—

Table 3 — Elements most common in animal; plant — and micro ortalisms (source not knwon)

**COLOURFUL CHEMICAL DEMONSTRATIONS (SEN 1982, Vol. 31 No. 2)**  
Dennis Mather, Leichhardt HS

**1. Rainbow**

Place 50mL of saturated boric acid solution (Note: boric acid dissolves slowly) in a 250mL beaker and stir with a magnetic stirrer. Add several drops of universal indicator so that the solution becomes bright red. Slowly add 0.5 mol L<sup>-1</sup> sodium hydroxide solution. The sequence of colours produced is red, orange, yellow, green, blue, indigo and violet. The sodium hydroxide solution must be added dropwise initially but once the solution becomes green it can be added more quickly. At the end, addition of acid drives the system

back to any of the above colours. This is a straight titration of saturated boric acid solution with aqueous sodium hydroxide, the universal indicator responding to the pH changes. Boric acid acts as a monobasic acid. For the addition of 50mL of sodium hydroxide, the pH change is approximately from 4 to 10. Students could be given the pH ranges for the various colours of universal indicator and asked to plot a pH/volume curve which should be that for a weak acid/strong base titration. The pH vs volume curve is shown in Figure 1.

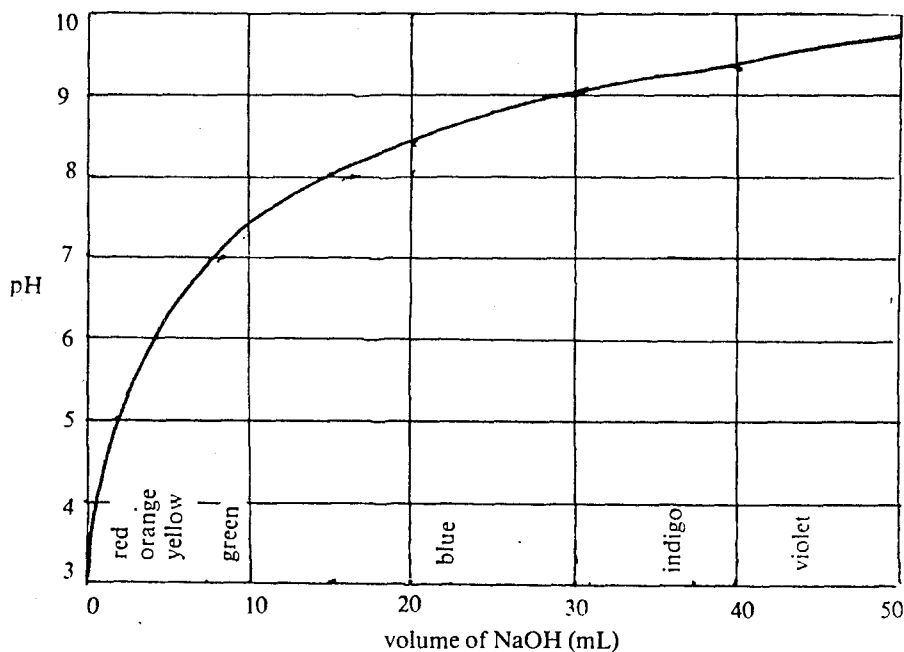


Figure 1.



## 2. Colour Changing Bottles

- (i) Dissolve 11g potassium hydroxide in 500mL of water in a florence flask and then add 18g of glucose. Add sufficient crystals of methylene blue to give a deep colour;  
or, 300mL water + 10g dextrose + 10g sodium hydroxide (the important thing is to have a reducing sugar in an alkaline solution).
- (ii) Repeat using indigo carmine instead of methylene blue.
- (iii) After dissolving the potassium hydroxide add a very few crystals of methylene blue and leave to stand until the solution turns purple before adding the glucose.

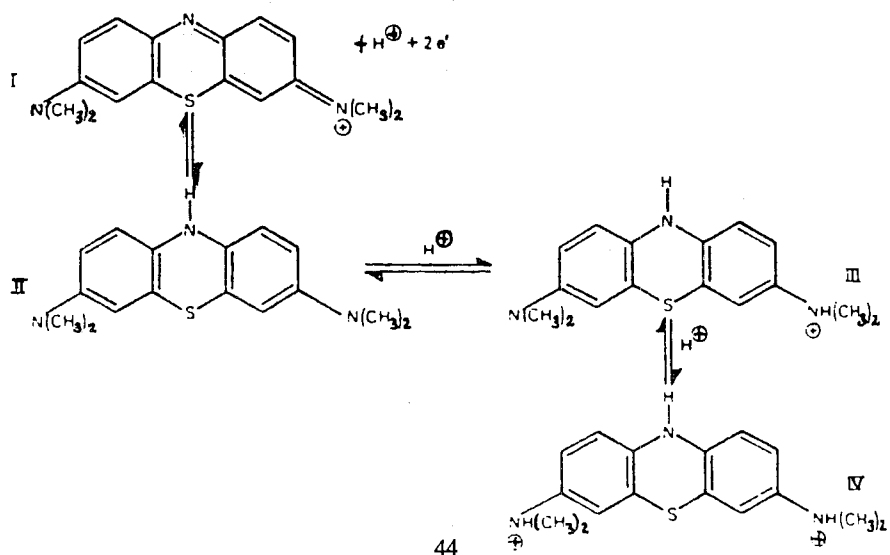
Stopper the bottles and allow to stand. Gently swirl and then vigorously shake the flasks.

- (i) The solution goes from colourless to blue.
- (ii) The colour changes from yellow, through red to green. An appropriate patter about traffic lights may be used.
- (iii) The solution goes from colourless to purple.

The original colours return on standing. When the colours fade, they can be rejuvenated by removing the stopper to let in air.

This demonstration is a very useful deductive exercise in kinetics, as shown in a stimulating discussion of this demonstration; the explanation given here takes a somewhat different view of the process. The reaction involves aerial oxidation of the glucose which, on standing, reverts to the reduced form, the colour changes being due to the redox indicators. The potential (relative to the saturated calomel electrode) varies from about 150mV on vigorous shaking to about 500mV on standing. Other redox indicators with appropriate potentials could be used. In the case of the indigo carmine, careful observation after vigorous shaking shows that the red appears initially at the bottom of the flask while for methylene blue a thin layer is visible in the meniscus, both observations providing evidence for aerial oxidation.

The redox chemistry of methylene blue has been extensively studied; it is complicated by the fact that methylene blue is sensitive to the pH. The currently accepted reaction scheme is as follows: at the pH of this demonstration the reactions involve species I and II.



### 3. Oxidation-Reduction Reactions Involving Transition Metal Ions

#### (i) Reduction of Vanadate with Zinc

Prepare a solution of vanadate ions by dissolving 10g of ammonia vanadate in 500mL 0.5 mol L<sup>-1</sup> sulfuric acid. Add granulated zinc to some of this solution.

The zinc will appear to be reacting with the acid in the usual way, but after about five minutes the colour of the vanadate solution will change. If it has not, add some extra acid, say 1mL 8 mol L<sup>-1</sup>.

Thus the vanadate ion is reduced by zinc (or atomic hydrogen) through recognisable stages from V (V) to V(II), if you are patient enough (possibly up to 30 minutes).

The species obtained are listed with their colours:

VO <sub>3</sub> <sup>-</sup> (aq)	orange	V <sup>3+</sup> (aq)	green
VO <sup>2+</sup> (aq)	blue	V <sup>2+</sup> (aq)	violet

The divalent ion is quite unstable and is easily oxidised to V<sup>3+</sup>(aq) by atmospheric oxygen. It will only be formed while there is plenty of zinc and acid in the solution. Also, fill the test tube so as to minimise the surface area/volume.

(ii) **Reduction of the Chromate Ion with Zinc**

This is a similar reaction to the one above. Prepare a solution of potassium chromate by dissolving 10g of potassium chromate in 500mL of 0.5 mol.L<sup>-1</sup> sulfuric acid. Add granulated zinc to some of this solution.

This reaction will produce Cr<sup>3+</sup><sub>(aq)</sub> with no trouble at all, in about 10 minutes. If a test tube full of the solution is used with an excess of zinc, the patient observer will obtain a glimpse of the Cr<sup>2+</sup><sub>(aq)</sub> ion.

The colours of the various species of chromium formed are given below:

CrO<sub>4</sub><sup>2-</sup><sub>(aq)</sub>      yellow

Cr<sup>3+</sup><sub>(aq)</sub>          green

Cr<sup>2+</sup><sub>(aq)</sub>          blue

**4. Cobalt (II) as an Indicator of Humidity**

Cobalt (II) is well known as an indicator for water, in self-indicating silica gel (for desiccating) and in cobalt chloride paper.

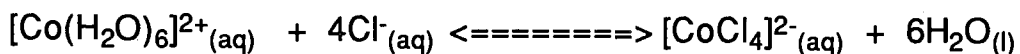
Cobalt chloride paper can be made by preparing a solution of 10g of cobalt chloride in 50mL of water. Soak filter paper in the solution and dry it in an oven until it turns blue. Store in a desiccator over a desiccant.

Similar colour changes can be produced in solution. Aqueous Co (II) is pale pink, but if crystals of cobalt (II) chloride are dissolved in ethanol the solution is an intense blue colour.

This can be used as an example of an equilibrium reaction since water added to the ethanol solution will turn it pink. It is, however, more difficult to turn the aqueous solution blue by adding ethanol; best conditions occur with very concentrated solutions in small quantities.

In water the cobalt exists as [Co(H<sub>2</sub>O)<sub>6</sub>]<sup>2+</sup>, an octahedral coordination compound. Each of the water molecules has formed a coordinate bond, through the oxygen, with the cobalt.

Cobalt (II) is capable of forming tetrahedral complexes under certain conditions. [CoCl<sub>4</sub>]<sup>2-</sup> is an intensely blue species and probably other tetrahedral complexes, such as [Co(H<sub>2</sub>O)Cl<sub>3</sub>]<sup>-</sup>. A simplified reaction can be written:

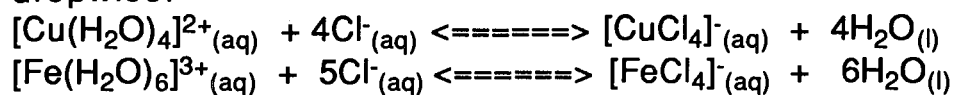


This reaction can also be investigated in aqueous solution, using concentrated hydrochloric acid as the source of chloride ions.

## 5. Colourful Equilibria with Copper (II) and Iron (III) Solutions

Prepare concentrated solutions of copper nitrate and iron (III) chloride. Since copper nitrate is deliquescent, the solution which often forms on the top of these crystals in reagent bottles is ideal for these purposes.

Add concentrated hydrochloric acid dropwise to 1mL of each of these solutions in separate test tubes. The copper (II) will change from blue to green/yellow; the iron (III) will change to a much paler yellow. These changes can be reversed by adding water dropwise.



**1. Introduction**

Towards the end of the eighteenth century chemists displayed a particular interest in the reactions between different gases. In 1781, Henry Cavendish found that whenever he exploded hydrogen with oxygen, a given volume of hydrogen would react with half its own volume of oxygen. In 1805, Joseph Louis Gay-Lussac repeated this experiment and confirmed that two volumes of hydrogen always reacted with one volume of oxygen, provided that the volumes of hydrogen and oxygen were measured under the same conditions of temperature and pressure.

Gay-Lussac was so intrigued by the simplicity of the 2:1 ratio that he went on to examine the combining volumes of other pairs of gases and found that:

1 volume hydrogen + 1 volume chlorine $\rightarrow$  2 volumes hydrogen chloride  
 1 volume nitrogen + 2 volumes oxygen $\rightarrow$  2 volumes nitrogen(IV) oxide

In 1808, Gay-Lussac published his Law of Combining Gas Volumes. When gases react, the volumes of the reacting gases and the volumes of any gaseous products are in the ratio of small whole numbers, provided the volumes are measured at the same temperature and pressure.

Chemists then began to look for an interpretation of Gay-Lussac's Law in terms of Dalton's Atomic Theory of Matter published in 1803. They realised that 100mL of nitrogen just reacted with 200mL of oxygen and the number of nitrogen and oxygen molecules must be simply related, otherwise some molecules of oxygen and nitrogen would be left over after the reaction.

In 1811, the Italian scientist Amadeo Avogadro suggested an important hypothesis which later became known as Avogadro's theory: equal volumes of all gases at the same temperature and pressure contain equal numbers of particles. This suggestion by Avogadro explains beautifully the experimental results which lead to Gay-Lussac's Law.

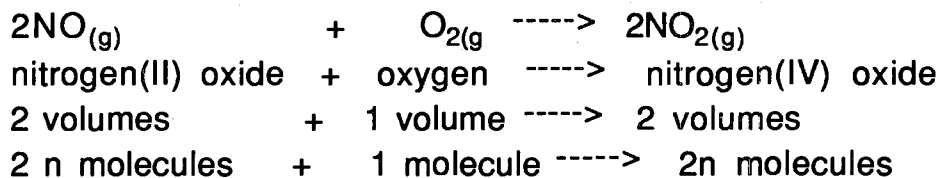
For example, in the reaction of nitrogen and oxygen if we assume one volume of nitrogen contains  $n$  molecules, then by Avogadro's theory two volumes of oxygen contain  $2n$  molecules and two volumes of nitrogen dioxide contain  $2n$  molecules.

nitrogen	+	oxygen	$\rightarrow$	nitrogen(IV) oxide
1 volume	+	1 volume	$\rightarrow$	2 volumes
$n$ molecules	+	$n$ molecules	$\rightarrow$	$2n$ molecules

In all reactions involving gases the number of moles of gas is proportional to its volume at constant temperature and pressure. There is no reason to believe that the total volume of reactants should equal the total volume of products because there is no rule concerning the conservation of the number of molecules in a chemical reaction.

Suitable reactions to demonstrate these theories in the school are difficult to find but the one described below provides for us the basis for a spectacular example.

Nitrogen(II) oxide (nitric oxide, NO) reacts with oxygen producing nitrogen(IV) oxide (nitrogen dioxide, NO<sub>2</sub>) as the only product.



Nitrogen(II) oxide is a colourless gas, a neutral oxide and sparingly soluble in water. Nitrogen(IV) oxide is a red-brown gas, soluble in water forming an acidic solution.

## 2. Procedure

Two demonstrations can be used to show the reaction.

### Demonstration 1

- Take two 500mL round bottom flasks. On each determine the level of 50 per cent volume. The usual 500mL round bottom flask occupies a volume of about 830mL from the base of an inserted cork. Therefore the 50 per cent volume mark will read 415mL.
- Set up the apparatus shown in Figure 1 and generate nitric oxide by slowly dropping concentrated sulfuric acid onto solid sodium nitrate covered with about three times its weight of water. This produces very pure nitric oxide. The reaction can be summarised by the following equation:



When water is left to the level of the base of the longer glass tubing in flask 1, clamp the rubber tubing at positions A and E. Disconnect the rubber tubing from the gas generator at A. This preparation should be performed in a fume cupboard.

## Figure 1. Generation of Nitric Oxide

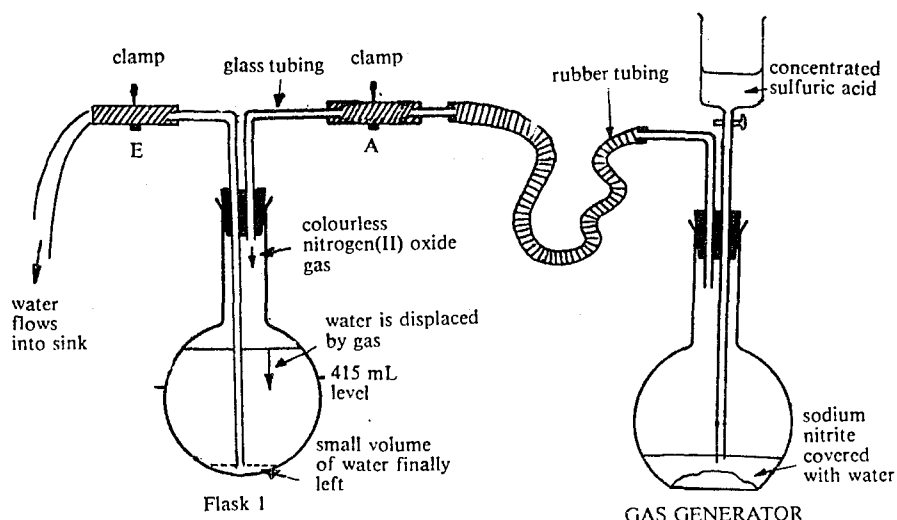
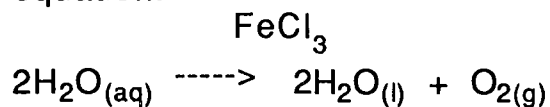


Figure 1. Generation of Nitric Oxide

- (c) Set up the apparatus shown in Figure 2 and generate oxygen gas by dropping 10 vol. hydrogen peroxide onto 20mL of a hot aqueous solution of iron(III) oxide until the water level is at the base of the longer glass tube. Ferric chloride acts as a catalyst in the decomposition of hydrogen peroxide according to the following equation:



Disconnect the rubber tubing at C, clamp the rubber tubing at position B and insert the glass tubing into the rubber tubing at position A on the flask containing the nitrogen(II) oxide.

- Note:**
- Generate oxygen gas for a minute or so in order to remove the original air in the gas generator.
  - Generate all gases with care. Add concentrated sulfuric acid and hydrogen peroxide slowly so a steady slow rate of gas generation is maintained.

**Figure 2. Generation of Oxygen Gas**

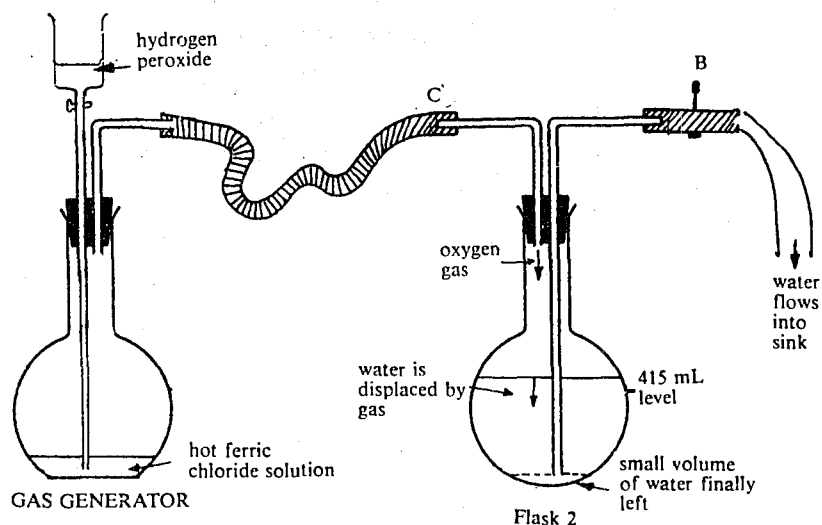


Figure 2. Generation of oxygen gas

- (d) Fill a large beaker with two litres of water, coloured green with universal indicator and connect by means of glass tubing to position B as shown in Figure 3. Both clamps are closed.

**Figure 3. Reaction of Nitrogen(II) Oxide with Oxygen**

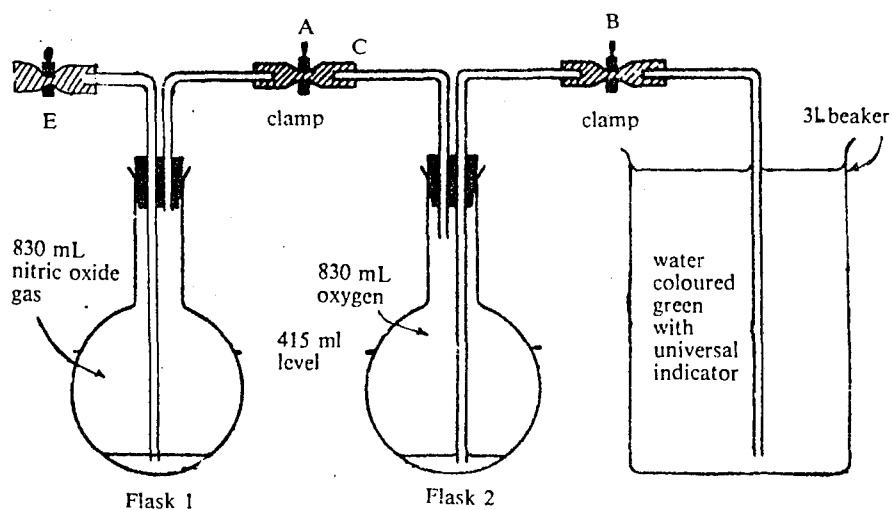


Figure 3. Reaction of nitrogen(II) oxide with oxygen.



- (e) The apparatus shown in Figure 3 is now ready. Open clamps A and B. After a minute or so the faint red-brown colour of nitrogen(IV) oxide appears at the opening to flask 1. This creates a small decrease in gas volume with a corresponding reduction in pressure. Oxygen begins to diffuse into flask 1 and water moves onto flask 2 to take its place. By this time the reaction between nitrogen(II) oxide and oxygen is quite rapid, further reducing the pressure. Water from flask 2 begins to move into flask 1, dissolving all the nitrogen(IV) oxide that forms. The universal indicator turns yellow in flask 1, due to the solution of nitrogen(IV) oxide, for example:

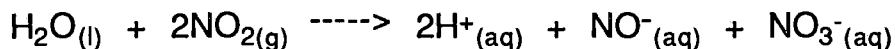
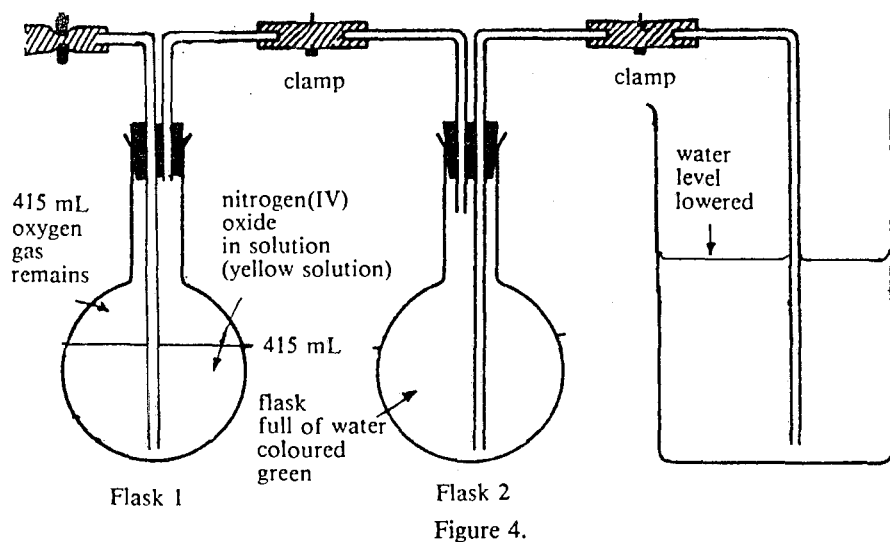


Figure 4 shows the state of the demonstration after complete reaction. The gas remaining in flask 1 can be tested. If nitrogen(II) oxide remains, combination with oxygen in the air on removing the cork forms brown nitrogen(IV) oxide. This does not occur. Oxygen is the remaining gas.

**Figure 4.**

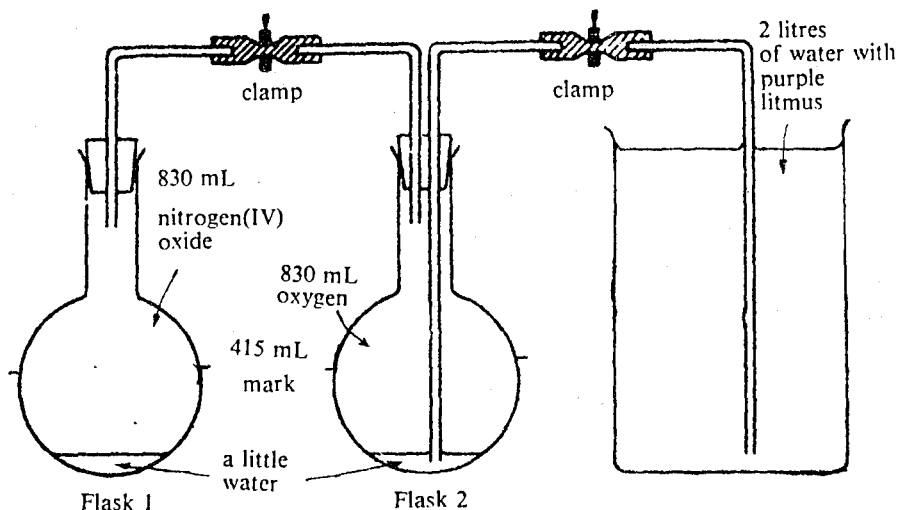


The demonstration clearly confirms that 830mL of nitrogen(II) oxide reacts with only 415mL of oxygen. It provides an ideal opportunity for careful observation and a worksheet could be designed to incorporate the chemistry involved.

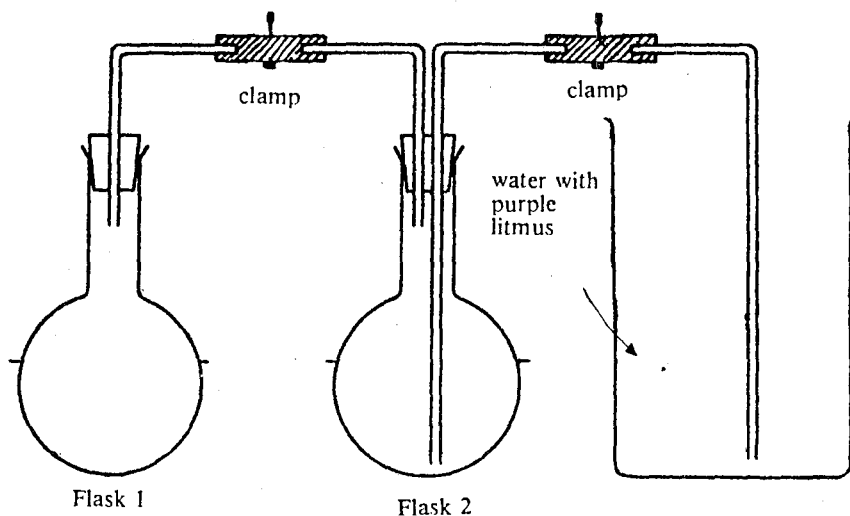
### Volume Relationships Between Reacting Gases

Nitrogen(II) oxide (NO) reacts with oxygen gas producing nitrogen(IV) oxide as the only product.

- (a) The apparatus below illustrates the initial stage of the demonstration. Both clamps are closed.

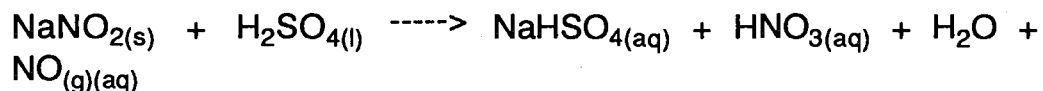


- (b) Both clamps are opened together. On the diagram below draw in any new water levels and record all your observations.



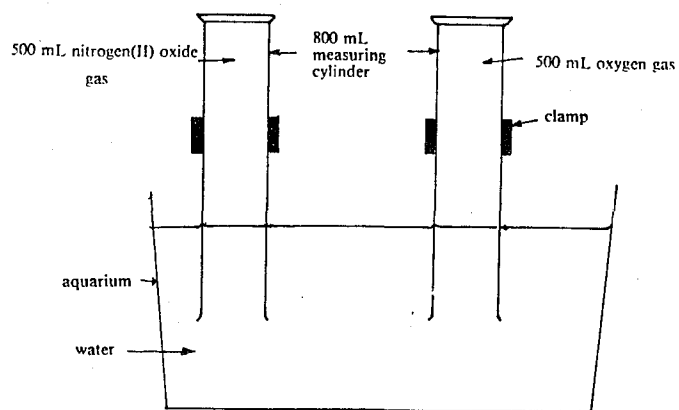
## WORKSHEET

1. Make an assumption about the nature of the remaining gas in flask 1. 2. Suggest a method to identify the gas.
3. The method in 2 was performed. Record the result and the nature of the remaining gas.
4. State the volume ratios that nitrogen(II) oxide and oxygen combine in.
5. What conclusion can you draw about the volume ratios of reacting gases from this demonstration.
6. Write a balanced equation for the reaction in which nitrogen(II) oxide combines with oxygen to form nitrogen(IV) oxide.
7. Water moves into the flasks from the beaker as a result of a reduction in pressure. Give a reason for this reduction in pressure.
8. Explain the change in colour of the universal indicator solution. Supplement your explanation with an equation.
9. The oxygen required for the demonstration was prepared by the decomposition of hydrogen peroxide using manganese as a catalyst. Write an equation for this reaction.
10. The nitrogen(II) oxide was prepared by the reaction of sodium nitrate and concentrated sulfuric acid shown by the reaction below. Balance the equation.

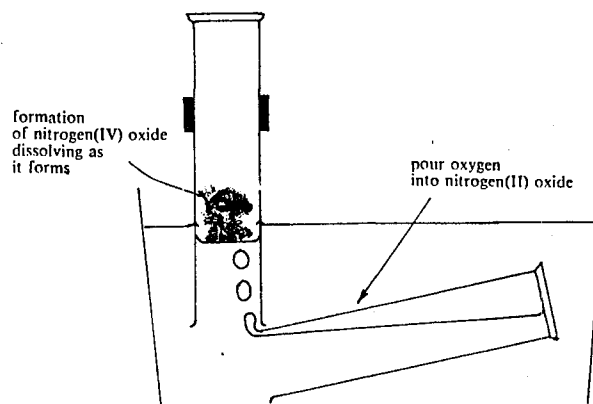


### Demonstration 2

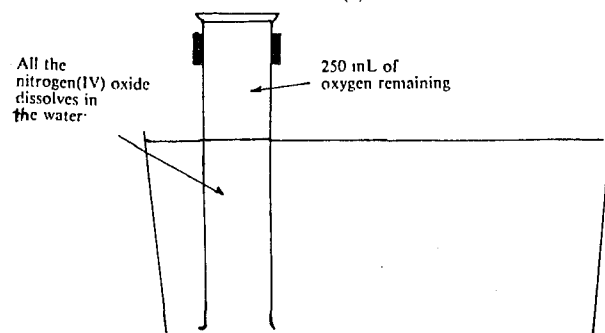
- (a) Set up the apparatus shown in Figure 5a. Into one of the measuring cylinders collect 500mL of nitrogen(II) oxide and in the other 500mL of oxygen. Adjust the cylinders so that the pressure of both gases is constant.
- (b) Pour all the oxygen gas slowly into the nitric oxide. The red-brown nitrogen(IV) oxide that forms dissolves, leaving 250mL of oxygen gas.



(a)



(b)



(c)

Figure 5.

Have you used colourful as well as smelly demonstrations to illustrate what you thought was the kinetic theory? The intent was to show that molecules in water or in air are in constant motion. If you had enough time in the period you would put dye into a beaker of water and have students watch the colour diffuse. On rushed days, however, you would put an open dish of ammonia water on the front desk and wait for the grimaces at the rear of the room.

Unfortunately neither demonstration illustrates diffusion, a process of *random* spreading in which particles of a liquid, gas, or solid collide spontaneously. In the case of ammonia in air, breezes and convection produce air currents that mechanically and *non-randomly* mix the air and ammonia. Similarly, ordered currents due to convection, density gradients and unsettled liquid are responsible for mixing the dye in the water. These demonstrations no more show diffusion than scrambling an egg illustrates 'diffusion' of the yolk.

Even bromine evaporating in a measuring cylinder does not really illustrate pure diffusion. The bromine vapour moves up the measuring cylinder, in part because it is mechanically pushed up by the almost thousandfold increase in volume that occurs in the change from a liquid to a vapour. As more liquid evaporates more gas is pushed up and more air is forced out of the measuring cylinder.

An experiment to show the rate of ammonia diffusion through air can be performed using a 50x1cm glass tube. The tube is supported horizontally and plugged with cotton. A few drops of concentrated ammonia solution are placed on the inside end of the cotton plug and a few drops of concentrated hydrochloric acid on the other. (The cotton plugs release the excess pressures and therefore eliminate mechanical forces on the gas.) In about 20 minutes a white disc of ammonium chloride forms where the two gases meet. As the experiment demonstrates, without much mechanical mixing the diffusion of ammonia gas is quite slow.

What about the dye demonstration in water? It takes several minutes of standing still before the large scale mechanical movements of the liquid cease; even then invisible fluid movements are caused by convection. To let students see these, tell them to do the following.

Fill a 250mL beaker with 4-5cm of soapy water made by adding several drops of dishwashing detergent to about 1L of hot water. Shake the beaker until there is a layer of foam on top, approximately 2cm deep,

made of small bubbles. Cover the beaker and let it stand for at least five minutes.

When all evidence of movement stops, carefully place two or three drops of food colouring on the top centre of the foam. In one to three minutes, the dye solution filters down between the bubbles and enters the liquid. (If not, add another drop or two of dye)

The foam hinders evaporation and cooling of the liquid and arranges the descending dye into an array of streams that slowly drain down. As long as the beaker is not moved, a complex and persistent pattern of colour slowly and mysteriously develops throughout the liquid, outlining the otherwise invisible convection cells that constantly transport fluid in an uncovered glass of water.

The shape of the pattern is dependent upon variables like the depth of the water, the shape of the glass, the temperature difference between air and liquid and even the proximity of the glass to a light or window. (The radiant energy from a closely held flashlight provides enough heat to visibly perturb the dye pattern.) The pattern stands out best when viewed against a white background such as a piece of paper.

## A GAS SAMPLING DEVICE (SEN 1982, Vol. 31 No. 4)

The Science Teacher: March 1982

Inflate a balloon with gas. Use only the volume of gas required by your class. The advantage of using a balloon is that it can be expanded to contain 'large' volumes of a variety of gases. Insert a 5cm length of plastic tubing (11-20mm diameter) into the neck of the balloon. Insert a rubber septum into the open end of the plastic tubing. Use a simple plastic syringe to remove samples of the gas for testing.

## A GAS DISCHARGE TUBE (SEN 1984, Vol. 33 No. 1)

Warwick Shephard, Macquarie BHS

A gas discharge tube can be easily assembled using government issue equipment. See Figure 1 for set up. It is important to note that wires A and B must be separated by at least 3 to 5cm and that wire A must be at least 7cm above the base of the bell jar apparatus (to avoid arcing).

This apparatus can illustrate such things as cathode and anode glow, negative glow and positive striations. It can also be used to demonstrate the relationship between gas discharges and changing pressures.

**Variation:** Wires A and B can be replaced by various shaped copper pieces to produce a larger glow area.

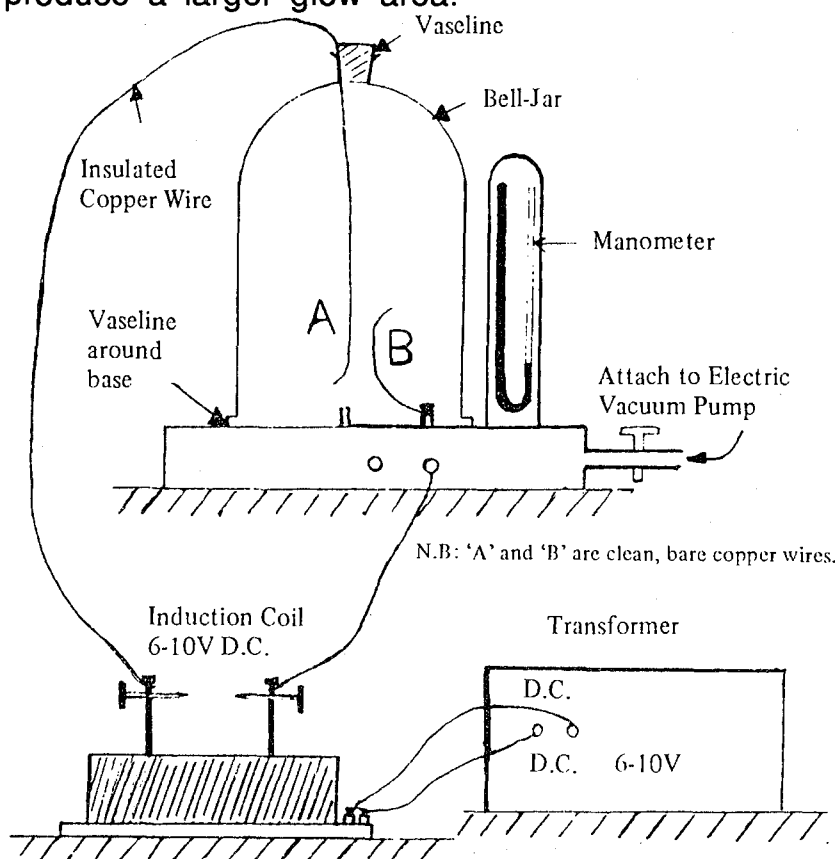


Fig. 1. Gas Discharge Tube Reprinted from Labscene, Vol. 7, No. 3 1983

## **SOAP MAKING - AN ALTERNATIVE RECIPE THAT WORKS (SEN 1983, Vol. 32 No. 3)**

Joy Reid, Peakhurst HS

### **Discussion**

The origin of soap is a mystery, but one suggestion is that Roman women discovered it by accident about 3000 years ago. They washed their clothes where rain water flowed from the hills into the River Tiber and over altars where animals had been slaughtered. The fat from the sacrifice and the ashes from the fire are said to have combined as the first crude soap.

### **Basic Recipe**

- 1/2 cup of cold water
- 2 tablespoons caustic soda
- 1 cup of melted tallow.

(Tallow can be made by melting lumps of animal fat (dripping) over a low heat in a heavy saucepan. Strain to remove streaks of blood and bits of meat, etc., if necessary. Place in warmed screw top jars and store in the refrigerator.)

### **Method**

Place the water in a heat resistant bowl, add caustic soda and stir immediately. When the caustic soda solution has cooled to lukewarm, add lukewarm melted tallow slowly, stirring continually. Beat gently with an egg whisk for about 4 minutes. (At this stage other fats and oils may be added to vary the soap, perfume and colour may also be added, whisking well) Pour the mixture into clean, *pliable* plastic moulds. (Margarine containers, pre-pack dessert containers, plastic trays for holding yoghurt, ice cube trays, etc.) and leave to set for about 24 hours. Gently ease the soaps out of their moulds and transfer onto a piece of kitchen paper to mature in a dry, well-ventilated place for two weeks.

### **Variations**

1. Some of the tallow in the above recipe may be replaced by other oils, e.g. coconut oil, olive oil, almond oil, castor oil, safflower oil, baby oil. The tallow seems to be what makes the soap hard, so the more oil in the mixture the softer the soap becomes. General rule of thumb; have at least 1/2 tallow 1/2 oil.
2. Perfumes; use essential oils, e.g. rose oil, lavender oil, pine oil, strangeflower oil, cinnamon oil or sandalwood oil (about 2 teaspoons). Also, rose water, witch hazel (1/4 cup, but reduce the amount of water in the recipe).
3. The soap can be coloured by adding food colourings.
4. Experiment with additions like infusions of ground herbs, oatmeal, lanolin, cucumber, avocado or strawberry pulp. (These are all reputed to be beauty treatments!)



### **1. Amoeboid Motions**

These are best performed on an overhead projector.

- \* Fill a petri dish with 4-6 molar ammonia solution and into the centre place a drop of oleic acid. The oleic acid will disperse rapidly into the ammonia solution, showing rapid amoeboid movements as it does so. More drops can be added until neutralisation occurs. The product is ammonium oleate.
- \* A second, more spectacular version uses mercury, nitric acid and potassium dichromate. Into a dilute solution of nitric acid add a drop of mercury about half the size of a 1 cent coin. Place a rice grain sized crystal of potassium dichromate into the petri dish. As the dichromate dissolves a yellow colour is seen to diffuse through the nitric acid. When the yellow boundary encounters the drop of mercury, rapid amoeboid motions result. These will continue for some time. Mention must be made that the movements observed are much more rapid than those an amoeba is capable of.

### **2. Nylon**

Prepare the following solutions:

- a) 1.5mL of adipyl chloride in 50mL of cyclohexane.
- b) 2.2g of 1, 6- diamiohexane and 4g sodium carbonate in 50mL of water.

Slowly add the adipyl chloride solution to the water solution.

Polymerisation will occur at the interface. Pull out the interfacial film with forceps and run it over a set of rollers. Once sufficient nylon has been formed, the system will be self sustaining.

The collected nylon should be washed in ethanol, then air dried at 110°C.

### **3. Squeezing a Glass Bottle**

Many of us experience discrepant events but hardly ever try to figure out, in a logical manner, why and how they occur. Here is an activity to stimulate interest in learning science through an activity which seems contrary to our first line of reasoning.

**Materials:** water, 10 cent coin, empty drink bottle.

**Procedure:** Place the coin over the mouth of the bottle. Drop a little water around the edge to seal it. Grasp the bottle with both hands and squeeze. The coin will begin to dance up and down. As the air inside the bottle expands from being warmed by your hands, the coin lifted to relieve the pressure. The squeezing per se has no effect upon the glass other than to raise the temperature.

## DEMONSTRATION DIAPHRAGM CELL FOR THE ELECTROLYTIC PRODUCTION OF SODIUM HYDROXIDE (SEN 1984, Vol. 33 No. 1)

This model cell is easily constructed from common materials and it shows in a convincing manner that hydroxyl ions are produced at the cathode and chlorine at the anode.

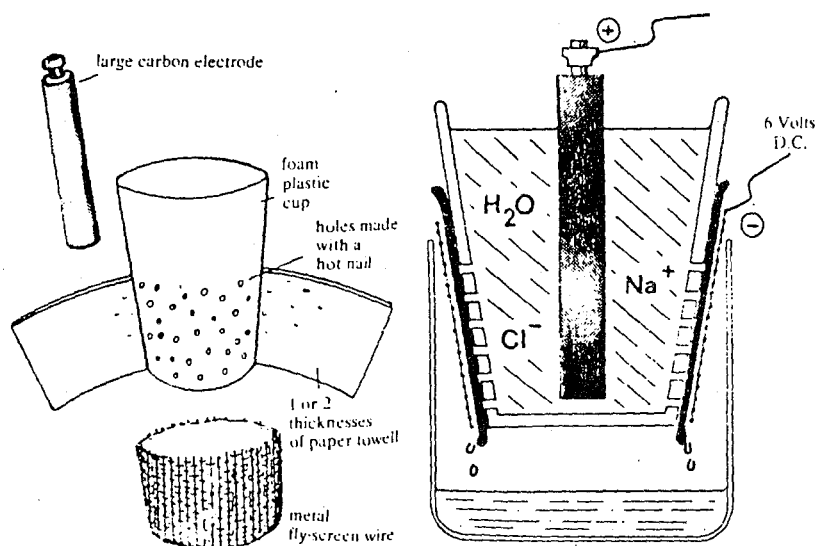
A foam plastic cup provides a porous container when holes, 1 or 2mm in diameter are made in the lower half of the cup and that part of the cup is wrapped in one or two thicknesses of paper towelling or filter paper. Metal gauze fly-wire provides a wrap around mesh cathode and the whole apparatus sits easily in a glass beaker. The carbon anode may be made from one or more rods taken from used dry cells, preferably large ones. The salt solution should be concentrated.

The production of hydroxyl ions at the cathode can be shown strikingly by adding phenolphthalein indicator to the salt solution or, alternatively, to the beaker which collects the solution which 'weeps' through the porous diaphragm.

Anode Reaction:  $2\text{Cl}^- \rightarrow \text{Cl}_2 + 2\text{e}^-$

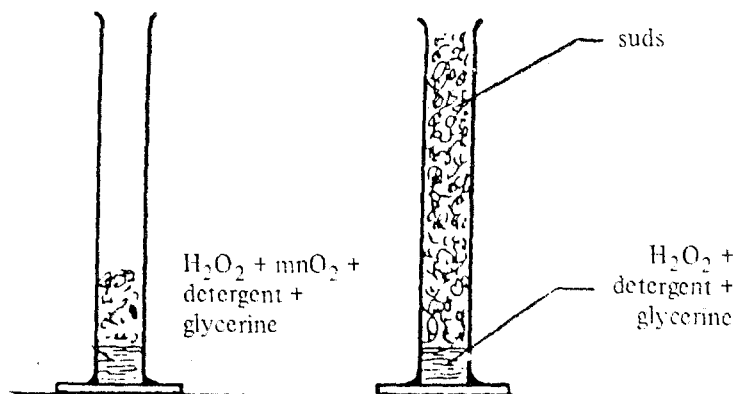
Cathode Reaction:  $2\text{H}_2\text{O} + 2\text{e}^- \rightarrow 2\text{OH}^- + \text{H}_2$

The 'secret' of this cell's practical operation appears to be in equating the rates of electrolysis with the rate of solution seeping through the permeable diaphragm. Naturally there is usually an appreciable quantity of the impurity of the sodium chloride in the product.



## Materials

1. Two tall cylinders or narrow jars.
2. Liquid detergent, glycerine, hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), manganese dioxide ( $\text{MnO}_2$ )
3. Two long stirrers (long straws will do).



## Procedure

1. Place about 10mL of hydrogen peroxide and 5mL of detergent and about 5mL of glycerine in each cylinder.
2. Add a pinch of  $\text{MnO}_2$  to one of the cylinders and stir the mixture in both cylinders with the long stirrers.
3. Observe the difference in height of the produced suds in the two tall cylinders.

## Questions

1. In which of the two cylinders did the suds rise higher?
2. What causes the suds to go up higher in one of the cylinders?
3. What do we need to make soap bubbles?
4. What function does the peroxide have in this reaction?
5. What do the  $\text{MnO}_2$  and the glycerine do?
6. Would the suds rise just as high without the stirring?
7. What other substances might be used in place of  $\text{MnO}_2$ ?

## Explanation

The detergent functions like the soap when blowing soap bubbles. By adding some glycerine to the detergent, the surface tension of the liquid is increased and the bubbles will stay longer before collapsing. In order to get suds in a soap solution when blowing bubbles, we need to blow through a straw into the detergent mixture. In this case we do not need to blow. Where are we getting the gas to blow the bubbles from? The gas is supplied by the hydrogen peroxide, which decomposes into water and oxygen. The  $\text{MnO}_2$  acts as a catalyst for this oxygen producing reaction. It does not partake in the reaction, but only facilitates it. This means that the properties of  $\text{MnO}_2$  before and after the reaction are unchanged. Dust and dirt particles or ashes could replace the  $\text{MnO}_2$ .

## MAKING A BREATHALYSER (SEN 1984, Vol. 33 No. 2)

Robyn Smith and Fred Love, Parramatta HS

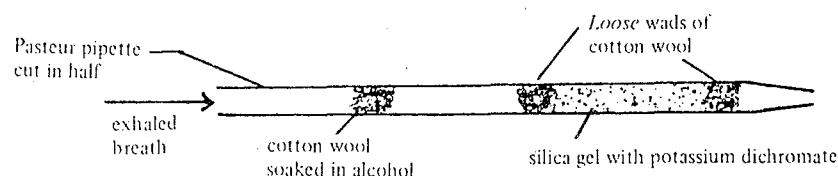
A breathalyser test kit can be made simply and quickly by students. The method below works well and can be used as a chemical experiment or to stimulate discussion on the social problems of alcohol and drink-driving.

### Materials

- \* Silica gel, chromatographic grade (as supplied)
- \* Potassium dichromate, as saturated solution in 3mol.L<sup>-1</sup> sulfuric acid
- \* 3-4mm tubing, e.g. plastic pasteur pipette
- \* cotton wool

### Procedure

1. (Optional). Heat the silica gel **gently** to drive off absorbed water. Cool to room temperature.
2. Cover a small quantity of silica gel (1-2g) with the dichromate solution. Pour off excess solution.
3. Spread the silica gel onto blotting paper or filter paper and move around with a spatula until dry.
4. Pack the tubing with the silica gel as shown below.



5. In the absence of any inebriated teachers (or students) (!) the action can be demonstrated by using a small wad of cotton wool soaked in ethanol. Breathe out through the tube for 10-15 seconds. The colour change in the 'crystals' takes 1-2 minutes to develop fully.

## **A. The Solubility of Potassium Chlorate**

### **Introduction**

Potassium chlorate is a slightly soluble salt. By measuring its solubility over a range of temperatures, the solubility product constant, ( $K_{sp}$ ), for potassium chlorate can be calculated over a range of temperatures.

### **Materials**

Electronic balance	potassium chlorate
5 test tubes	test tube rack
bunsen burner	tripod
gauze	500mL beaker
thermometer	10mL measuring cylinder
spirit based marking pen.	

### **Procedure**

1. Prepare a beaker of boiling water.
2. Label 5 test tubes 1 to 5 and add 1.00g, 1.25g, 1.50g, 1.75g and 2.00g respectively of potassium chlorate to the test tubes. (These weights are not critical; if you find you have 1.02g, say, in test tube 1, leave it, but record **that** weight in your book.)
3. Add 10.00mL of water to each test tube and place them in the boiling water bath. Swirl gently until all the solid dissolves.
4. Remove test tube 1 and place a thermometer in it. Observe the test tube for the first sign of crystallisation and **note** the temperature.
5. Repeat with test tubes 2 to 5.

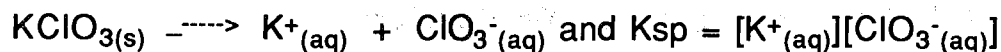
Record your results as indicated below.

Test tube	Weight of $KClO_3$	Temperature of Saturated Solution
1	1.00g	
2	1.25g	
3	1.50g	
4	1.75g	
5	2.00g	

Graph the weight of  $KClO_3$  versus the temperature of the saturated solution.

### Calculations

The equation for the dissolution of  $\text{KClO}_3$  is:



In this experiment the solubility of  $\text{KClO}_3$  in moles per litre (M) is

$$[\text{K}^+_{(aq)}] = [\text{ClO}_3^-_{(aq)}]$$

1. Calculate the solubility of  $\text{KClO}_3$  in moles per litre for each of your five results.
2. Using these results calculate the value of  $K_{sp}$  for each temperature where crystallisation first occurred.

### Question

Is the dissolving of  $\text{KClO}_3$  an exothermic or an endothermic process? Explain, using Le Chatelier's Principle.

### B. The Solubility Product of Silver Chromate

This experiment enables the range of values for the  $K_{sp}$  of silver chromate to be determined.

#### Materials

$1 \times 10^{-2} \text{ mol L}^{-1} \text{ AgNO}_3$  (0.425g/250 mL)

$1 \times 10^{-4} \text{ mol L}^{-1} \text{ K}_2\text{CrO}_4$  (0.486g/250mL then a 1 in 100 dilution)

6x150mL beakers, 3 measuring cylinders, 2 gradual pipettes.

#### Procedure

Label 6 beakers 1 to 6, then add  $\text{H}_2\text{O}$ ,  $1 \times 10^{-2} \text{ mol L}^{-1} \text{ AgNO}_3$  and  $1 \times 10^{-4} \text{ mol L}^{-1} \text{ K}_2\text{CrO}_4$  to each as indicated in the table below

	Beaker Number					
	1	2	3	4	5	6
$\text{H}_2\text{O}$	40mL	60mL	70mL	80mL	85mL	90mL
$1 \times 10^{-2} \text{ mol L}^{-1} \text{ Ag}^+$	40mL	20mL	10mL	10mL	10mL	5mL
$1 \times 10^{-4} \text{ mol L}^{-1} \text{ CrO}_4^{2-}$	20mL	20mL	20mL	10mL	5mL	5mL

Where a precipitate occurs (cloudiness) the ion product  $> K_{sp}$  and where no precipitate occurs the ion product  $< K_{sp}$ . Hence the range of  $K_{sp}$  values can be determined.

Typically beakers 1, 2 and 3 are cloudy and beakers 4, 5 and 6 are clear. This indicates a  $K_{sp}$  value of about  $1 \times 10^{-11}$  to  $2 \times 10^{-11}$ .

### C. Precipitation, a Case of Equilibrium

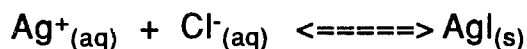
A student demonstration

#### Materials

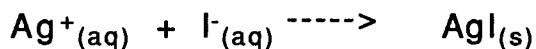
0.1 mol L<sup>-1</sup> NaCl, 0.1 mol L<sup>-1</sup> AgNO<sub>3</sub>, 0.1 mol L<sup>-1</sup> KI, 1 mol L<sup>-1</sup> KI, 2 test tubes, stirring rod.

#### Procedure

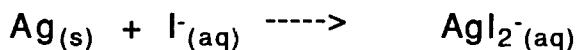
1. Add 2mL of 0.1 mol L<sup>-1</sup> NaCl to 2mL 0.1 mol L<sup>-1</sup> AgNO<sub>3</sub> to produce a white precipitate of AgCl.



2. Then add 2mL of 0.1 mol L<sup>-1</sup> KI while stirring. Note that the white colour of AgCl disappears and the yellow colour of AgI forms.



3. Then add 1mol L<sup>-1</sup> KI with stirring; note that the AgI dissolves.



#### Questions

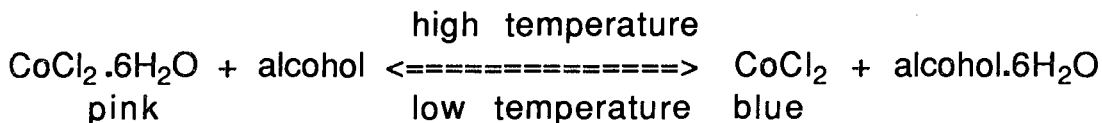
1. Why does the AgCl disappear?
2. Which is the more insoluble salt, AgCl or AgI?
3. Explain the observations in terms of Le Chatelier's Principle.

**SOLAR ENERGY STORAGE: A DEMONSTRATION** (SEN 1984, Vol. 33 No. 3)

H.S. Kimmel and R.S. Tompkins; Journal of Chem. Ed. Vol. 56, No. 9, SCP 1979.

The concept of storing solar energy using phase change materials is receiving considerable attention at the present time. The inclusion of a simple demonstration experiment to illustrate this idea in general or physical chemistry provides an excellent example of the application of thermochemical principles. Several phase change materials are being considered, such as sodium sulfate decahydrate (Glauber's salts), where the stored energy is represented by the heat of hydration.

A laboratory experiment to determine the heat of hydration of a salt hydrate has been suggested. However, for the purposes of a lecture demonstration this experiment is unsuitable due to the time available and also the effect is not particularly visual. A more convenient demonstration experiment which is effective in showing the use of a phase transition for heat storage uses the reaction between cobalt chloride and alcohol.



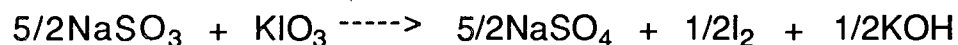
The demonstration is performed as follows. Dissolve about 1g of cobalt chloride hexahydrate,  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$  in 10mL of isopropanol so as to obtain a deep blue coloured solution. Water should then be added dropwise until a distinct pink colour appears. The water molecules preferentially solvate the cobalt ion forming the pink salt hydrate. The solution is then placed in a test tube or flask and heated in a beaker of hot water (solar energy). Almost immediately the pink colour is replaced by a blue solution as the anhydrous cobalt chloride has now formed. The pink colour will appear again as soon as the solution is immersed in cold water (cold night). This process can be repeated indefinitely.



The reactions discussed here are based on the famous clock reaction, which uses  $\text{KIO}_3$  and  $\text{NaHSO}_3$ . If sufficient  $\text{NaHSO}_3$  is present the reaction will proceed as follows:



There will be no change in appearance. When little  $\text{NaHSO}_3$  is present the reaction proceeds thus:

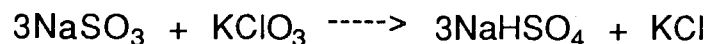


In the above reactions, suitable concentrations of the solutions are 0.05M  $\text{KIO}_3$  and 0.125M  $\text{NaHSO}_3$ . These two solutions will be referred to as A and B respectively. In the following reactions, these are diluted by about half. If A and B are equal in volume, the mole ratio will be 2.5:1; the subsequent reaction will be the clock reaction and  $\text{I}_2$  will become visible in about one minute.

If the volume of the  $\text{NaHSO}_3$  solution is 1.2 times that of the  $\text{KIO}_3$  solution, the mole ratio becomes 3:1, and the first reaction above will result. In practice, good reliability is achieved by using a volume of  $\text{NaSO}_3$  solution more than 1.3 times the volume of the  $\text{KIO}_3$  solution.

**Note:** the concentrations should be those stated above unless otherwise specified.

In the following reaction,  $\text{KClO}_3$  may be used instead of  $\text{KIO}_3$ . In this case, the reaction proceeds as follows:



There will be no change in the appearance of the solution. A suitable concentration of  $\text{KClO}_3$  is 0.4M. This will be referred to as solution C.

## TRICK 1

### Method

Dilute 30mL of A with 110mL of water and add 6mL of 0.1N solution of  $\text{HgCl}_2$ . Then add 45mL of B and mix continuously using a magnetic stirrer. The solution will first appear colourless and transparent, but as  $\text{I}^-$  is produced through reaction of A and B, gradually  $\text{Hgl}_2$  suspension

will appear as an orange suspension. As  $I^-$  increases further, the  $HgI_2$  suspension will then change to the complex ion  $HgI_4^{2-}$  and the solution will become colourless and transparent as before. Note: when too much  $HgCl_2$  is added the suspension will not disappear completely.

Caution is advised in the use of  $HgCl_2$  because of its toxicity.

### **Alternate Method**

To 30mL of A, 100mL of C and 7.5mL of a 0.1N solution of  $HgCl_2$ , add 45mL of B and mix continuously using a magnetic stirrer. The solution, which is colourless and transparent at first, will gradually become a muddy orange. After some time, it will suddenly turn a dark brown. If a suitable quantity of starch is added beforehand, it will turn a deep purplish blue. If the amount of  $Hg^{2+}$  is less, the colour of the  $I_2$  will appear after the suspension disappears.

### **TRICK 2**

#### **Method**

Dilute 30mL of A with 30mL of water and add 90mL of a 0.1N solution of  $Pb(NO_3)_2$ . Then add 45mL of B. A dense white suspension will form. Mix continuously using a magnetic stirrer. Since  $I^-$  is produced by reaction of A and B, the superfluous  $Pb^{2+}$  reacts with  $I^-$  and becomes  $PbI_2$  and the suspension will turn yellow. As mixing is continued,  $SO_4^{2-}$  is yielded and  $PbI_2$  will change to  $PbSO_4$ , which is a less soluble substance than  $PbI_2$ , so that the suspension will turn white again.

**Note:** when there is too little  $Pb^{2+}$ , in spite of the presence of  $I^-$ ,  $PbI_2$  will not be formed and the suspension will not turn yellow. On the other hand, if there is too much  $Pb^{2+}$ , in spite of the presence of  $SO_4^{2-}$  all the  $PbI_2$  will not turn into  $PbSO_4$  and the yellow colour will not disappear completely.

### **TRICK 3**

#### **Method**

Mix 30mL of B (1M), 45mL of a 2N solution of KI and 75mL of C. The resulting solution will be an almost colourless (slightly yellowish) solution. Let the solution stand for a while and it will gradually change from yellow to bright yellow. The colour will subsequently fade away. If the solution is allowed to stand for a while longer, it will begin to turn yellow again and finally will become re-orange. The last change will take place more rapidly if the solution is heated. The colour changes occur when the mole ratio of  $H_2SO_3$  to  $I^-$  is in the range of 1:4.

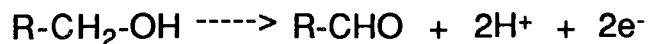
# AN OXIDATION REACTION: 1-PROPANOL TO PROPANOL

(SEN 1985, Vol. 34 No. 2)

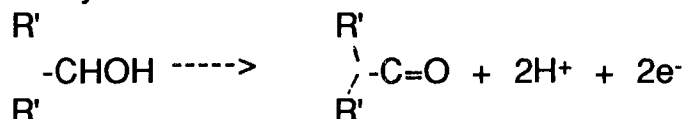
Chris Wiecek, Warilla HS

## Introduction

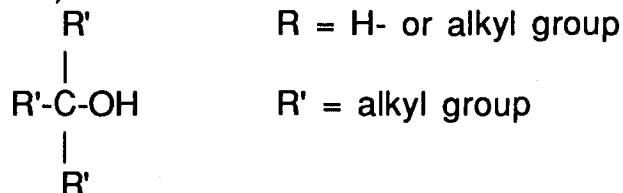
Alcohols differ in their behaviour with oxidising agents. Primary alcohols are oxidised first to an aldehyde and then to a carboxylic acid.



Secondary alcohols are oxidised to ketones.



Tertiary alcohols are not oxidised with oxidising agents other than oxygen. (They will burn.)



In the case of primary alcohols the oxidation of the aldehyde formed is easier than its formation, hence it is difficult to isolate the intermediate aldehyde. However, aldehydes usually have much lower boiling points than either alcohols or carboxylic acids, so that it is sometimes possible to distil out the aldehyde from the reaction mixture as it is formed.

For example: propanol has B.P. = 97°C  
propanal has B.P. = 49°C  
propanoic acid has B.P. = 141°C

If the reaction mixture for the oxidation of propanol is maintained at 85-90°C only propanol will distil out.

Typical oxidising agents suitable for oxidation of alcohols are acidified solutions of permanganate or dichromate salts of sodium or potassium.

## Equipment and Chemicals

1-propanol (n-propanol, propyl alcohol), conc. sulfuric acid, sodium dichromate,  
0.1 mol L<sup>-1</sup> silver nitrate, 0.1 mol L<sup>-1</sup> potassium dichromate,  
0.1 mol L<sup>-1</sup> aqueous ammonium solution.

### Quickfit

50mL pear shaped flask, stillhead, condenser, tap funnel, thermometer, teflon adaptor, receiver adaptor, Claisen stillhead, fractionating column, boiling chips, clean test tubes, beakers, bunsen, clamps, retort stands, measuring cylinders.

### Procedure

Dissolve 10.5g of sodium dichromate in 20mL water. Cautiously add 8mL conc. sulfuric acid with stirring. Place 8mL propanol in a pear shaped flask and assemble the equipment as set out below.

Run water through the condenser. If a Claisen stillhead and fractionating column are available they attach to the top of the pear shaped flask, with the stillhead and condenser attached to the top of the fractionating column.

Add boiling chips to the propanol and place the acidified sodium dichromate solution in the tap funnel. Using a bunsen burner bring the propanol to boiling, then add the acidified sodium dichromate **dropwise** over about ten minutes. It should not be necessary to heat the reaction any further. The propanol will distil out of the reaction mixture. Note the boiling points above.

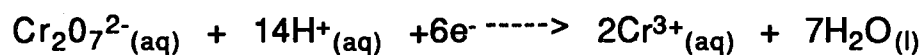
Collect the propanol in a test tube. A good yield is 2-3mL.

### Reactions of Propanol

1. Prepare Tollens reagent by adding 0.1mol L<sup>-1</sup> aqueous ammonia solution to 0.5mL 0.1mol L<sup>-1</sup> AgNO<sub>3</sub> dropwise until the precipitate just dissolves.  
Add 3 drops of propanol, warm (in a warm water bath). Observe.  
**Clean this test tube immediately**, as explosive substances may form on standing.  
A silver nitrate mirror will form on the glass, as the propanol is oxidised.
2. Add a drop of dilute sulfuric acid to 0.5mL 0.1mol L<sup>-1</sup> potassium dichromate. Add 3 drops of propanol and warm.  
The colour changes to green as the propanol is oxidised.
3. Add 3 drops of propanol to 0.5mL Benedicts or Fehlings solution.  
The colour changes to the red colour of Cu<sub>2</sub>O as the propanol is oxidised.
4. Repeat 1, 2 and 3 with propanol. What differences are there?

## Questions

1. Write the half-equation for the formation of propanol.
2. By combining it with the half-equation for the reduction of dichromate:



write the overall equation for the reaction.

3. 8mL of propanol was used in this experiment but only about 2-3mL of propanol was isolated. Suggest what may have happened to the 'missing' 5-6mL.

## **THE PREPARATION OF BENZOIC ACID FROM BENZYL CHLORIDE**

(SEN 1986, Vol. 35 No. 1)

Chris Wiecek, Warilla HS

Benzoic acid may be prepared from benzyl chloride under alkaline oxidation conditions. A substitution reaction occurs initially to form benzyl alcohol which oxidises to benzaldehyde and then to benzoic acid.

**N.B.** Care must be taken to avoid any contact with the skin with benzyl chloride and the refluxing must be done in a flask less than half full to avoid 'bumping'.

Benzyl chloride is available in many schools and hence this reaction provides a useful oxidation reaction for a chemistry class to observe carboxylic acid being produced. I do the experiment as a demonstration with class participation.

### **Materials**

500mL round bottomed flask, condenser, benzyl chloride, potassium permanganate, sodium carbonate decahydrate, sodium sulfate solution (20% w/v), conc. hydrochloric acid.

### **Procedure**

Place sodium carbonate decahydrate (10.8g), potassium permanganate (9g) and 200mL of water in a 500mL round bottomed flask. Add boiling chips and benzyl chloride (4.5mL). Reflux for 90 minutes, allow to cool, add conc. HCl (40mL) and then sodium sulfate while shaking until the manganese dioxide is dissolved and only a white precipitate of benzoic acid remains. Filter the benzoic acid off, wash with cold water and recrystallise from boiling water. The melting point is 120-122°C.

**HYDROLYSIS OF THE ESTER METHYL SALICYLATE (SEN 1985,  
Vol. 35 No. 1)  
Chris Wiecek, Warilla HS**

The hydrolysis of esters is part of the Carbon Chemistry elective in 2U Chemistry. The hydrolysis of methyl salicylate is a simple procedure where the products are dramatically distinct from the reactants. Methyl salicylate (present on most school shelves) is highly aromatic. Most students immediately recognise the odour. The products are methanol and salicylic acid (a white solid).

**Materials**

methyl salicylate, sodium hydroxide, conc. hydrochloric acid, boiling chips, 100mL round bottomed flask, condenser, stillhead, thermometer, teflon adaptor, fractionating column, acidified potassium dichromate solution.

**Procedure**

Weigh 20g of methyl salicylate into 100mL round bottomed flask. Add a solution of sodium hydroxide (6g in 40mL water) and boiling chips. Reflux until a clear solution results. Allow to cool and attach a fractionating column, stillhead, condenser and thermometer. Heat slowly and collect the fraction boiling below 70°C (B.P. of methanol is 65°C).

Test the product with an acidified solution of potassium dichromate. A small sample can also be tested for flammability. Finally, dismantle the glassware and acidify the solution in the round bottomed flask with conc. HCl (10mL). Allow to cool and filter off the salicylic acid. Wash with a little cold water and recrystallise from boiling water. Filter and dry the product and measure its melting point (158°C).

Dissolve a sample in water and test with blue litmus.

## **USING A DISSECTING MICROSCOPE IN TEACHING INTRODUCTORY CHEMISTRY (SEN 1986, Vol. 35 No. 3)**

Robert Winokur, University of Nevada; Manus Monroe, Indian Valley Colleges.

In an effort to have high school students develop observational skills and acquire an excitement about chemistry, stereoscopic dissecting microscopes (magnification 7 to 40X) have been used to observe the physical characteristics and chemical reactions of several substances, a few of which are described in this article. The reactions must be viewed using a bright source of direct incident (not transmitted) light. Both lights and microscopes can often be borrowed from a biology department. Depression slides are useful but not essential. Safety glasses are recommended, especially when working with acids.

### **Reactions with Copper Sulfate**

Students are instructed to adjust the magnification of their microscope to between 20 and 30X and place a drop or two of distilled water onto the depression of a glass slide. A few grains of freshly prepared anhydrous copper sulfate are placed, using a spatula, extremely close to, but not on the water drop. Students first observe that what appears as white powder to the naked eye is actually granular. Within 2-5 minutes, some of the granules gradually change to light blue crystals. After viewing the colour change, students are then instructed to push several granules into the water, using either a needle or a small glass rod and to observe the individual hydrated crystals. Students then add additional anhydrous copper sulfate to the drop of water and stir and the rate at which the crystals dissolve is noted. The relationship between the rate of dissolution and the concentration of a solution is discussed, as well as the concept of (equilibrium) vapour pressure of water and the use of copper(II) sulfate as an indicator in dessicants.

With the magnification set at 10 or 15X, a few particles of magnesium powder are finally added to the solution. The production of copper metal on the surface of the magnesium particles is then observed. The details and the possible mechanism for this redox reaction are discussed, as well as the physical contact required between the solid magnesium particles and the aqueous copper(II) ions in the solution. If the students see the production of gas bubbles (hydrogen) on the surface of the metallic particles, due to a redox reaction between magnesium and hydrogen ions, it would indicate that the distilled water was slightly acidic.

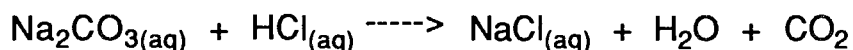


### **Reaction Between Magnesium Powder and 1 mol L<sup>-1</sup> HCl**

Another procedure that is especially effective for this type of magnified observation is the reaction between magnesium powder and 1 mol L<sup>-1</sup> HCl. The individual particles are animated by the production of hydrogen gas bubbles on their surfaces and make a compelling spectacle when viewed under magnification. Students are instructed to adjust the magnification to 10 or 15X, to place a few drops of 1 mol L<sup>-1</sup> HCl into the depression in the slide and to add a few particles of magnesium. A magnification of 20 to 30X will allow a close-up view of the animated particles and gas production. At this point, if appropriate, discuss the concept of redox reactions between hydrogen ions and some elemental metals.

### **Reactions Between Sodium Carbonate and HCl**

Students are instructed to adjust the magnification to between 10 and 30X. Next, they place approximately 1mL of 3 mol L<sup>-1</sup> sodium carbonate on a watch glass and slowly add, drop by drop, 1 mol L<sup>-1</sup> HCl to the solution. Students will observe the evolution of gas from the solution. Here, the typical reaction of an acid on a carbonate, producing carbon dioxide, may be discussed.



Similar reactions can be carried out with small pieces of chalk and crushed sea shells.

### **Reactions Between Copper Metal and Silver Ions**

Students are instructed to adjust the magnification to 10 or 15x and using a small piece of sandpaper or emery cloth, sand a 1-2cm piece of 12 or 14 gauge copper wire. This process removes any thin layer of oxides or chlorides and exposes pure wire. They then place a few drops of 1 mol L<sup>-1</sup> silver nitrate in the depression of the slide and place a 0.5cm piece of wire in the solution. After students have viewed the growth of needles and feather-like crystals, again a discussion of redox reactions between metallic atoms and cations of different elements may be appropriate.

### **Dissolving Potassium Permanganate in Distilled Water**

This procedure is designed to allow students to observe crystals dissolving and to note the local colouring around a dissolving crystal. Any magnification between 10 and 30X may be used. Again, students place a few drops of distilled water in the depression of the slide and using a spatula, place one or two potassium permanganate crystals in the water.

These few experiments serve to illustrate how direct observations of chemical reactions with a microscope can be used to teach and

dramatise basic chemical principles. Many similar experiments can be developed to suit the needs of individual courses. After all, chemistry is an experimental science. Without excitement in the laboratory in the skills of observation, students may fail to understand and appreciate our discipline.

Journal of Chemical Education 62, 157 (1985)

We were recently contacted for assistance by a distraught parent whose child's shirt was spotted with black colloidal silver following an encounter with silver nitrate solution.

We supplied her with the ingredients of Farmer's Reducer and she subsequently reported a 100% success rate in removal of the offending stain. Her experience supported our own testing with this reagent for removal of colloidal silver from textiles.

Farmer's Reducer has been used since time immemorial in photography to correct over-exposed negatives. It consists of two solutions:

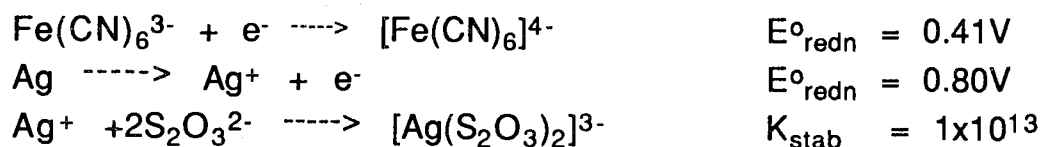
Solution A: Potassium ferricyanide,  $K_3Fe(CN)_6$  - 7.5% W/V

Solution B: Sodium thiosulfate,  $Na_2S_2O_3 \cdot 5H_2O$  - 25% W/V

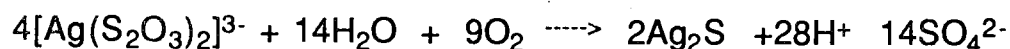
For stain removal equal amounts of the two freshly prepared solutions are mixed and applied to the material. The black colloidal silver dissolves and can be rinsed from the material. Thorough rinsing is essential in order to avoid the formation of black silver sulfide at a later stage.

In our test work we coated cheese cloth in 0.1M silver nitrate solution for five minutes. The cloth was removed and allowed to stand in sunlight for three weeks, at the end of which time it was uniformly stained to a delicate shade of black. A liberal application of Farmer's Reducer removed approximately half the stain within ten minutes and all of it within an hour.

The chemistry behind the process is the oxidation of silver metal to silver ion by the ferricyanide and the subsequent reaction of this ion with thiosulfate to give a soluble complex:



Incomplete rinsing of the material gives silver sulfide:



While the reduction potential of the ferricyanide reaction would appear to be too low to effect oxidation of metallic silver to soluble silver ion, the high stability constant for the thiosulfate silver complex ensures that the concentration of silver ion in solution remains low. Hence the reduction potential for the silver reaction is much lower than the  $E^\circ$  value and the oxidation of metallic silver to silver ions will proceed.

For those who are theoretically inclined, the following calculation can be carried out.

Assume that 1g of metallic silver is present and that the volume of Farmer's Reducer is one litre.

	Moles Present	
	Initially	After Oxidation
Ag	0.01	0
Ag <sup>+</sup>	0	0.01
[Fe(CN) <sub>6</sub> ] <sup>3-</sup>	0.11	0.10
[Fe(CN) <sub>6</sub> ] <sup>4-</sup>	0	0.01

For the complexing reaction, from the value of  $K_{stab}$  we can calculate

	Moles Present	
	Initially	After Oxidation
S <sub>2</sub> O <sub>3</sub> <sup>2-</sup>	0.5	0.48
Ag <sup>+</sup>	0.01	4.3x10 <sup>-15</sup>

Applying the Nernst Equation

$$E_{redn} = E^\circ_{redn} - \frac{0.059}{n} \log \frac{[\text{reduced species}]}{[\text{oxidised species}]}$$

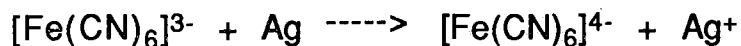
to the ferricyanide reaction,  $[\text{Fe(CN)}_6]^{3-} + e^- \rightleftharpoons [\text{Fe(CN)}_6]^{4-}$

$$\begin{aligned} E_{redn} &= 0.41 - 0.059 \log \frac{0.01}{0.1} \\ &= 0.47V \end{aligned}$$

and for the silver reaction,  $\text{Ag} \rightarrow \text{Ag}^+ + \text{e}^-$

$$\begin{aligned} E_{\text{redn}} &= 0.80 - 0.059 \log \frac{1}{4.3 \times 10^{-15}} \\ &= -0.05\text{V} \\ E_{\text{ox}} &= +0.05\text{V} \\ \text{Total } E &= E_{\text{redn}} + E_{\text{ox}} = 0.52\text{V} \end{aligned}$$

Thus the total reaction



will proceed.

Under normal circumstances the amount of metallic silver present per litre of solution will be far less than 1g and concentrations of Farmer's Reducer can be reduced but the reaction is slower. We found that a tenfold reduction in concentrations was still satisfactory, but the material required overnight soaking for complete removal of the silver stains. Even a 100 fold dilution performed adequately but two overnight soakings were required with fresh reagent being necessary for the second contact.

### Precautions

Sodium thiosulfate solution is not stable over long periods and so should be freshly prepared.

Farmer's Reducer decomposes rapidly. The two solutions must be mixed together only immediately before use.

Farmer's Reducer will react with some dyes. Test the solution on an unimportant area before use and, if necessary, use a diluted solution.

Following stain removal the material must be thoroughly rinsed to remove the thiosulfate silver complex and unreacted reagents.

### References

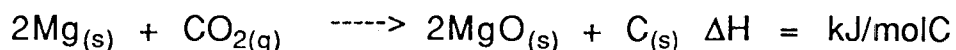
1. Farmer, H. 'Yearbook of Photography', p59 (1884), from C.B. Neblette, 'Photography, Its Principles and Practice', van Nostrand (1938), p365.

## BURNING MAGNESIUM IN DRY ICE (SEN 1988, Vol. 37 No. 3)

Mark Cox, Cranbrook School

### Introduction

A spectacular reaction which could be used to introduce oxidation/reduction material is the burning of magnesium in dry ice.



The reaction is extremely exothermic and should be carried out in a well ventilated area, well away from students. For the best effect the reaction should be done in a darkened room. The magnesium burns with an intense white flame and gas 'clouds' are forced out through the joint between the two blocks of dry ice. When the reaction has ceased, (20-30 seconds), a mixture of white MgO and black C can be observed.

1. Obtain two blocks of dry ice and create a pair of matching grooves, (at least 4cm).
2. Place a handful of Mg filings into the groove of the bottom block.
3. Wind approximately 30cm of clean Mg ribbon into fuse and insert it into the Mg filing pile.
4. Ignite the fuse, using a bunsen burner with a long rubber tube and then cover with the matching block.

**Caution:** Do not attempt the demonstration without having practised the procedure yourself. Inexperience could be potentially dangerous. Keep students well clear of the demonstration.

### Discussion Question

Why have CO<sub>2</sub> fire extinguishers been barred from racing tracks since the introduction of Mg alloy car wheels?

